Measurement and Analysis of Ambient Light Hydrocarbons — Montreal 1982

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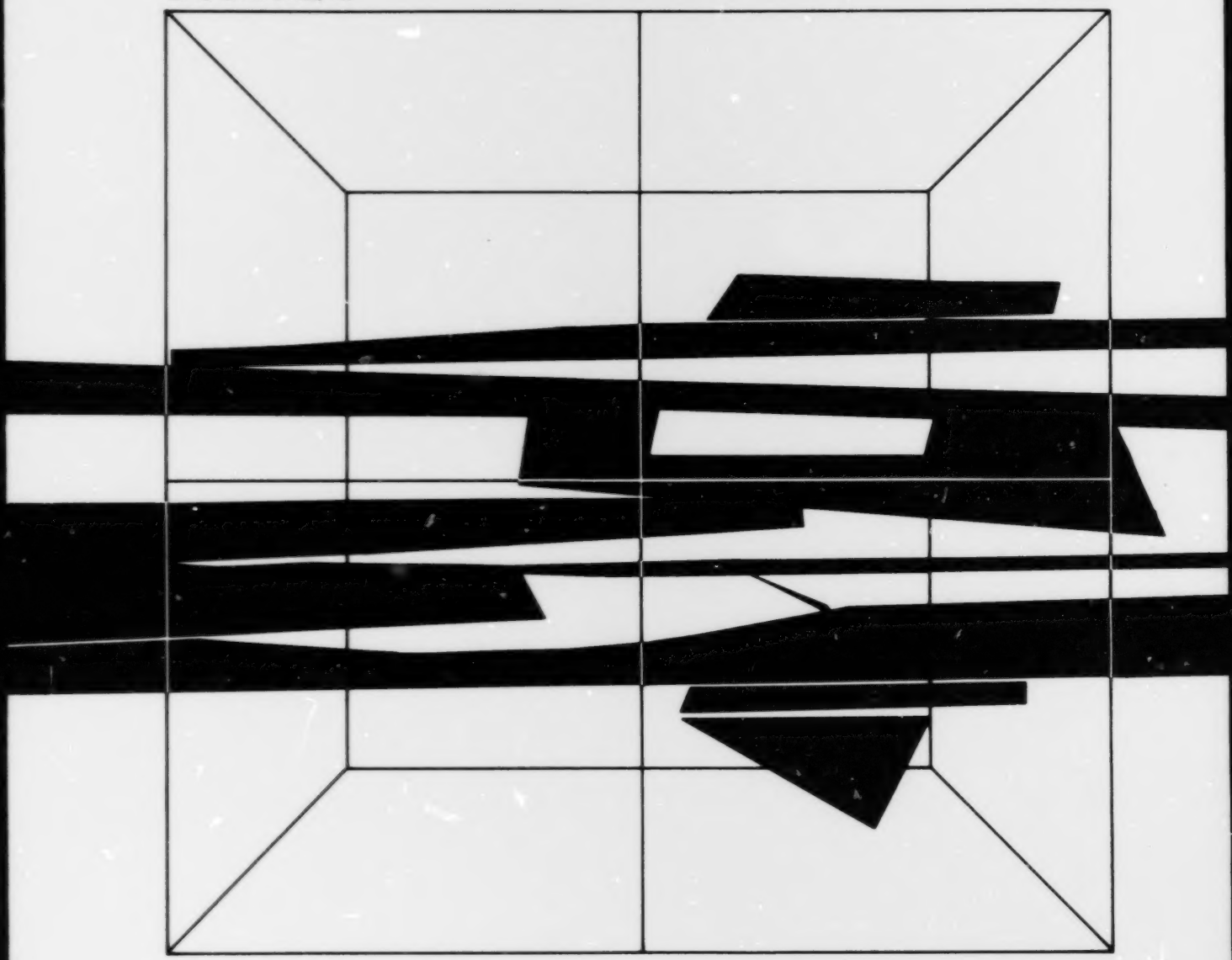

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MEASUREMENT AND ANALYSIS OF AMBIENT LIGHT HYDROCARBONS MONTRÉAL - 1982

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Report EPS 5/AP/RQ 1E
Mars 1985

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Minister of Supply and Services
Cat. No. En 49-5/5-RQ1E
ISBN 0-662-13838-4

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ABSTRACT

This project, to measure the concentration of ambient light hydrocarbons (C_2-C_6) in Montreal, is one of the activities of the Regional Office of Environment Canada, aimed at assessing and describing the problem of oxidants and their precursors in Quebec. The results of the study have laid the groundwork for a further study to examine the whole problem of oxidants in Quebec.

Samples for the study were taken in two separate areas of Montreal. The first sampling site (St Jacques) was located in a typically urban area, in downtown Montreal, where there is considerable traffic; the second (St Jean Baptiste) was in an industrial zone containing oil refineries.

The samples, taken over three-hour periods, were kept in Tedlar bags, and then transferred to a stainless steel canister. They were then analysed by a contractor, by means of gas chromatography, to determine their content in light hydrocarbons (c_2-C_6).

The average light hydrocarbon content measured at the St Jacques site (downtown) was 500 ppb C, and 600 ppb C at the St Jean Baptiste site.

RÉSUMÉ

Ce projet de mesure de la teneur en hydrocarbures légers (C_2-C_6) de l'air ambiant de Montréal faisait partie des activités du Bureau régional d'Environnement Canada visant l'évaluation et la caractérisation du problème des oxydants et de leurs précurseurs au Québec. Les résultats de cette étude ont servi de données de base à une étude subséquente visant à définir la problématique des oxydants au Québec.

Au cours de ce projet, des échantillons ont été prélevés dans deux zones distinctes de Montréal. Le premier poste (Saint-Jacques) était situé dans une zone typiquement urbaine, au centre de Montréal, où la circulation automobile est élevée; le second (Saint-Jean-Baptiste) était situé dans une zone industrielle caractérisée par la présence de raffineries de pétrole.

Les échantillons composés sur une période de trois heures ont été prélevés dans des sacs "Tedlar", puis transférés dans un cylindre en acier inoxydable. La composition en hydrocarbures légers (C_2-C_6) de ces échantillons a été déterminée par chromatographie en phase gazeuse.

Une teneur moyenne de 500 ppb C en hydrocarbures légers a été mesurée au poste Saint-Jacques (centre-ville) tandis qu'elle est de 600 ppb C à la station Saint-Jean-Baptiste.

1 INTRODUCTION

In recent summers, it has been noted at ambient air quality monitoring sites in Montreal and Quebec City that ozone levels regularly exceed the levels set as acceptable in the national air quality objectives. Ozone is a powerful oxidant, and its effects on vegetation are well documented.

We do not know for certain how ozone, considered the main oxidant, is formed. We do know, however, that two groups of substances, nitrogen oxides and hydrocarbons, are involved in the process and are considered important precursors of ozone. The transportation sector is a major source of nitrogen oxides (NO_x) and hydrocarbons (HC); in 1976, it accounted for 54.7% of total NO_x emissions and 39.1% of total HC emissions in Canada.

In order to assess and describe the problem of oxidants and their precursors in Quebec, the Regional Office of the Environmental Protection Service of Environment Canada conducted a study with a view to measuring light hydrocarbons (C_2-C_6) in ambient air in two separate districts of Montreal. The study took no account of methane (CH_4) levels, given the minor role this hydrocarbon plays in the generation of ozone. The results of the study laid the groundwork for a further study intended to examine the whole problem of oxidants in Quebec.

A total of 59 samples and 6 blanks were taken between August 31 and December 2, 1982. The meteorological conditions relating to each of the samples are described in Appendix I.

The three-hour integrated samples were collected in Tedlar bags, with a capacity of approximately 100 litres, and then transferred into a stainless steel canister at a pressure of about 375 KPa (40 psig). They were then analysed by a contractor, by means of gas chromatography, to determine their light hydrocarbon (C_2-C_6) content.

In this report we will discuss the sampling and analysis methods used and the results of the sampling.

2 SAMPLING

2.1 Sites

The choice of a sampling site for the measurement of hydrocarbons is an important factor, particularly in zones where there are several sources. The site must be several hundred metres away from major sources of hydrocarbons (gas stations, dry cleaners, surface coating operations and refineries), so as to provide for an acceptable degree of homogeneity. It must also be representative of a given geographic area.

For the purposes of this study, two sites were chosen, in two different zones. The first was in a typically urban zone, in downtown Montreal, where traffic is heavy; the other was located in an industrial zone near a number of oil refineries.

The two sites are part of the MUC ambient air quality monitoring network: the St. Jacques site (12) in downtown Montreal and the St. Jean Baptiste site (03) in Pointe aux Trembles (see map in Figure 1).

The St. Jacques site is located on the third floor of a building belonging to the City of Montreal, at 1125 Ontario East, at the corner of Amherst. The sampling manifold was placed on the roof of the building, about 18 metres from ground level.

The St. Jean Baptiste site was located in a shelter at 1050 St. Jean Baptiste Boulevard, in Pointe aux Trembles, to the northeast of the Montreal East petrochemical complex. The sampling manifold was placed about 4.8 metres from ground level. Note that there was a service station about 200 metres to the west of the shelter.

2.2 Equipment used

The following equipment was used for the sampling:

1. Pumps - So as to avoid any contamination, three different pumps were used:

   - Sampling pump: MB-21 stainless steel metal bellows pump.

   - Transfer pump: MB-158 stainless steel metal bellows pump, used to transfer the sample from the bag to the canister.

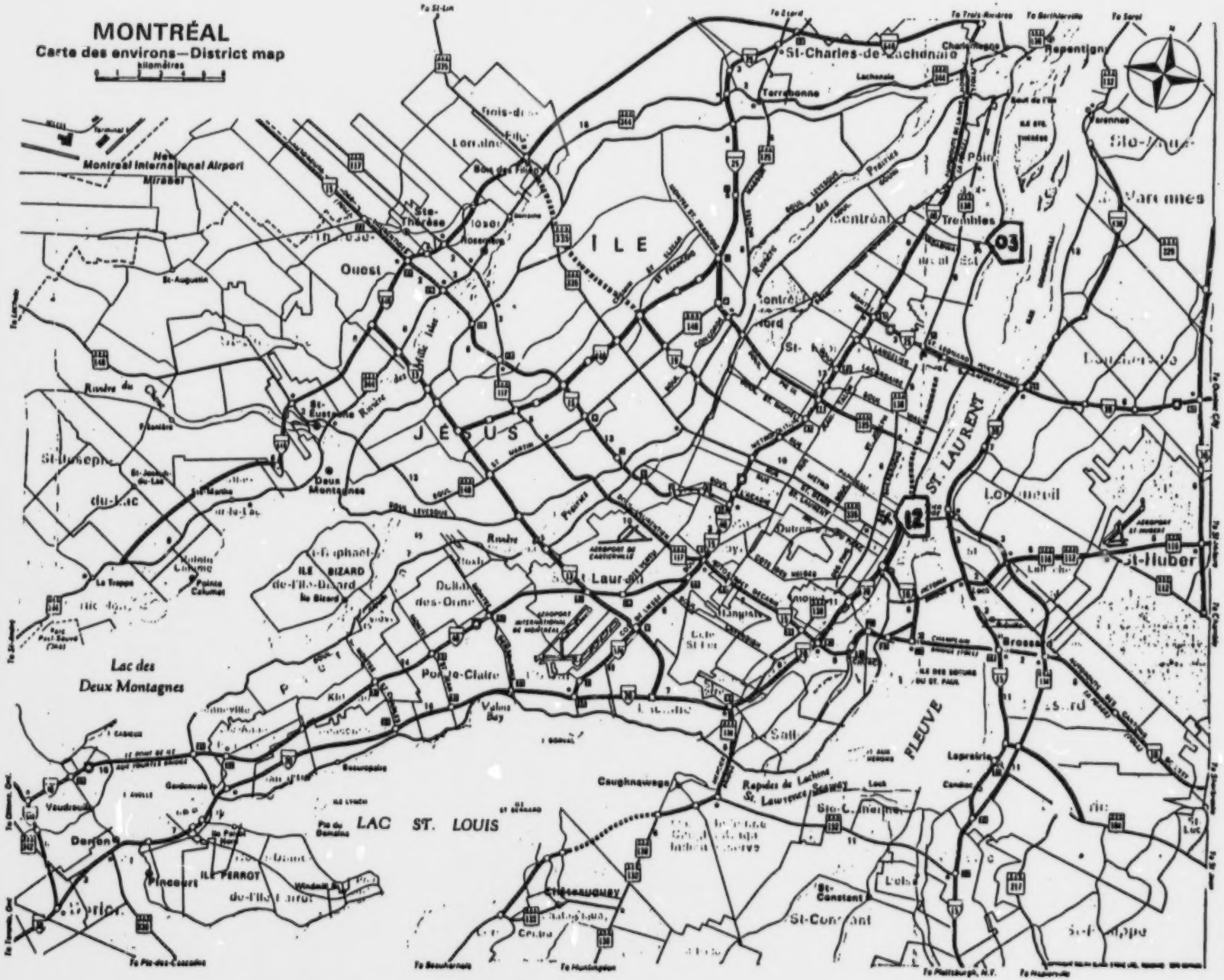


Figure I: Emplacement des sites d'échantillonnages

- Evacuation pump: MB-158 stainless steel metal bellows pump, used to flush the bag and canisters.

2. Bags - Tedlar bags with a capacity of up to 100 litres, and equipped with two valves, were used to collect the samples.

3. Flowmeter - A Mathescn 8160 model flowmeter was used to measure the flow of air pumped into the bag. A micrometric valve at the outlet of the sampling pump made it possible to control the flow precisely.

4. Canisters - The air samples were transferred into 3.78 litre (1 gallon) stainless steel canisters with valves at each end. The samples could be kept for up to a week in these canisters.

5. Heating tape - Required for cleaning the canisters.

6. Tubing and fittings - All the tubing used was of teflon (1/4") and the fittings of stainless steel (Swage lock).

2.3 Sampling procedures

2.3.1 Preliminary operations. The Tedlar bags and the canisters had to be thoroughly cleaned prior to the sampling, so as to avoid any contamination, as follows.

1. Canisters

The heating tape was wrapped around the canister, which was heated until its outside temperature reached about 100°C (this took approximately one hour).

- The canister was then flushed with ultrapure air at a rate of 500 ml/min. for about 30 minutes.

- Next, it was filled with ultrapure air at a pressure of 375 to 440 KPa (40-50 psig).

- Finally, it was allowed to cool before being filled with the sample.

Two canisters were usually connected in series and cleaned together so as to save time and gas.

2. Bags

- The bags were cleaned individually on the site, just before sampling was conducted.

- The bag was filled with ultrapure air.

- The bag was then flushed, with the evacuation pump.

- This procedure was repeated at least five times.

Immediately after the sample was transferred, the bag was flushed twice with ultrapure air.

2.3.2 Sampling. (See sampling diagram, Figure 2)

- The manifold was connected to the sampling pump.

- The pump was turned on and the flow adjusted. For a three-hour integrated sample, the flow was set at 500 ml/min. so as to collect a sample of 90 litres of air. This was quite sufficient to fill two 3.78 litre (1 gallon) canisters, including flushing.

- After about 10 to 15 minutes, the pump air outlet was connected to a cleaned and flushed Tedlar bag, and the starting time noted.

- Three hours later, the sampling pump was stopped and the valve on the bag closed.

- With the evacuation pump, the canister was evacuated to obtain the greatest vacuum possible (see transfer diagram, Figure 3).

- The Tedlar bag was connected to the intake of the transfer pump, and the outlet of the pump attached to the canister.

- The sample was then transferred into the canister, at a pressure of about 200 KPa (15 psig). The canister was then flushed with the evacuation pump. This operation was repeated twice to ensure that any residual air in the canister was the same composition as that in the bag. Finally, the canister was filled with the sample at a pressure of about 375 KPa (40 psig).

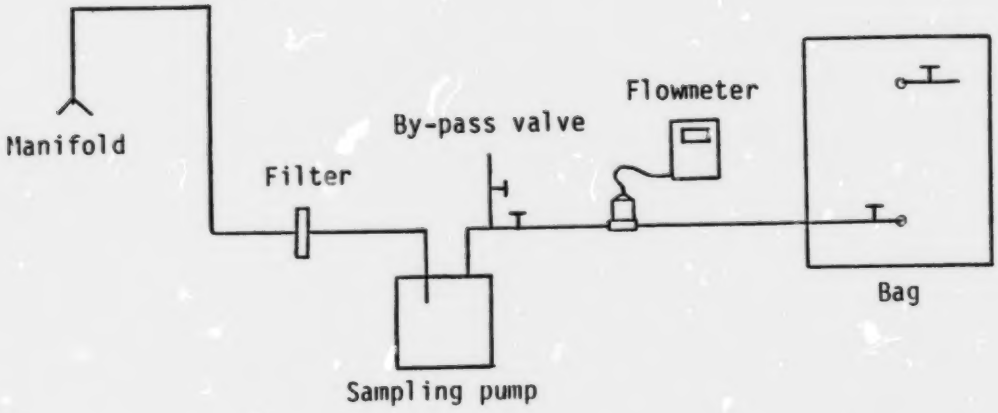


FIGURE 2

Sampling procedure

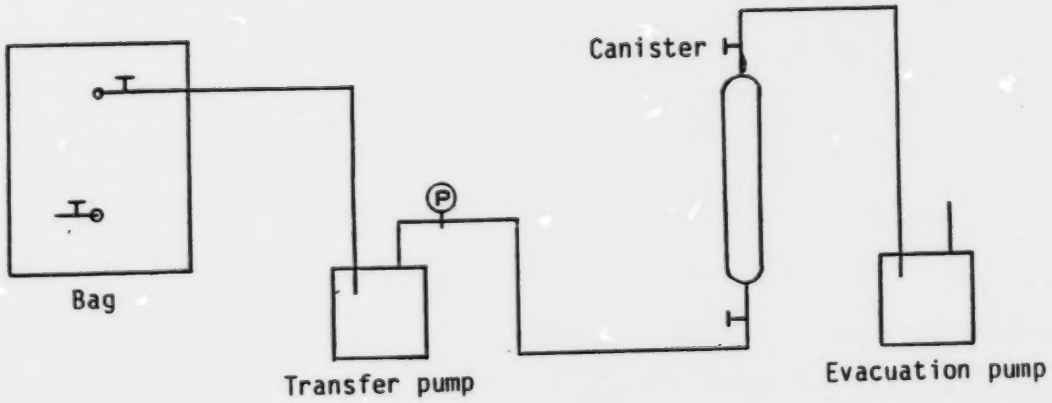


FIGURE 3

Transfer procedure

- The canister was then sealed with stainless steel plugs at both ends.

- It was labelled with the following information:

  - sample number
  - sampling site
  - canister number
  - date
  - time sample taken

3 ANALYSIS

3.1 Analysis method

The samples were analysed by gas chromatography with a flame ionization detector (GC-FID) to determine their light hydrocarbon content.

First, the samples were preconcentrated. A certain amount of the sample was injected into a freezeout loop (in liquid oxygen - BP: 183°C), where the hydrocarbons condensed and were captured on absorbent material. Because of the conditions during this preconcentration, methane was not captured. The trap was then immersed in boiling water and a carrier gas (helium) transferred the hydrocarbons to the chromatography machine.

3.1.1 Equipment used. The following equipment was used during the analysis procedure:

- Hewlett Packard 5750 chromatography machine, equipped with a 6 m (20 foot) x 0.317 cm (1/8 inch) i.d., Nickel, Durapack Phenyl Isocyanate/Porasil C, 80/100 mesh column. The following operating conditions applied during the analysis of light hydrocarbons (C_2-C_6):

- Oven: isothermal, 45°C

- Detector: flame ionization
  Hydrogen: 35 ml/minute
  Air: 500 ml/minute
  Auxiliary helium: 25 ml/minute

- Carrier gas: helium, 30 ml/minute

- Six-port sampling valve equipped with a cryogenic trap. The latter is a stainless steel loop, 10 cm (4 inches) long and with an interior diameter of 0.3175 cm (1/8 inch), packed with 80/100 mesh glass beads which act as absorbent material.

- 3.78 l stainless steel canister of the same type as those used during sampling.

- Precision pressure gauge (0-30 psi, scale 0.1 psi).

- Dewar vacuum flask and liquid oxygen.

- Hot plate.

- 1000 ml beaker with distilled water.

- Hewlett Packard 3390A reporting integrator.

The setup for sample injection is shown in Figure 4. Figure 5 shows the setup for calibration.

3.1.2 Determination of volume sampled. The volume injected must be known as precisely as possible in order to determine the hydrocarbon content. The volume was determined by means of the change in pressure.

A canister, of the type used during the sampling and of known volume (V_1), was placed at the exit of the freezeout loop. The volume of air from the sample was collected in this canister after passing through the freezeout loop. The volume sampled can thus be determined by means of the formula below.

$$V = \frac{298\ V_1\ (\Delta P)}{760\ (T + 273)}$$

where V = Volume of the sample at standard conditions (760 mm Hg and 25°C).

ΔP = Pressure difference (given by PG2, Figure 4) between the start (P_0) and completion (P_1) of the passage of air through the trap (mm HG) (P_1-P_0).

V_1= Volume of the canister.

T = Temperature of the sample in °C.

The volume (V_1) of the canister was determined by the difference in pressure corresponding to the volume of distilled water displaced, in a volumetric flask in a water bath, by the helium removed from the cylinder. In determining the average over four tests, it was found that V_1 was equal to 3.86 litres ±27 ml (if the pressure gauge reading was assumed to be ±0.05 psi).

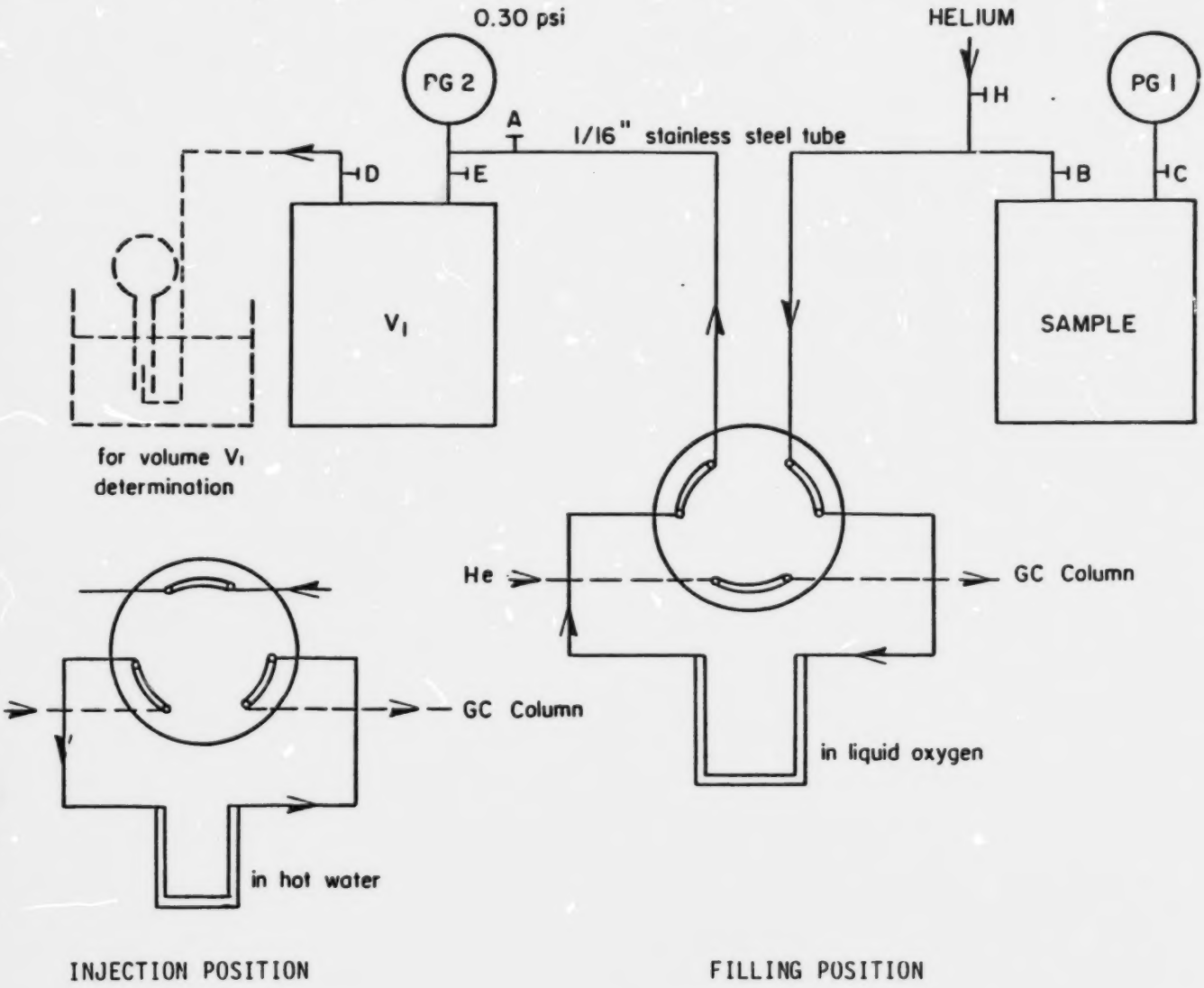
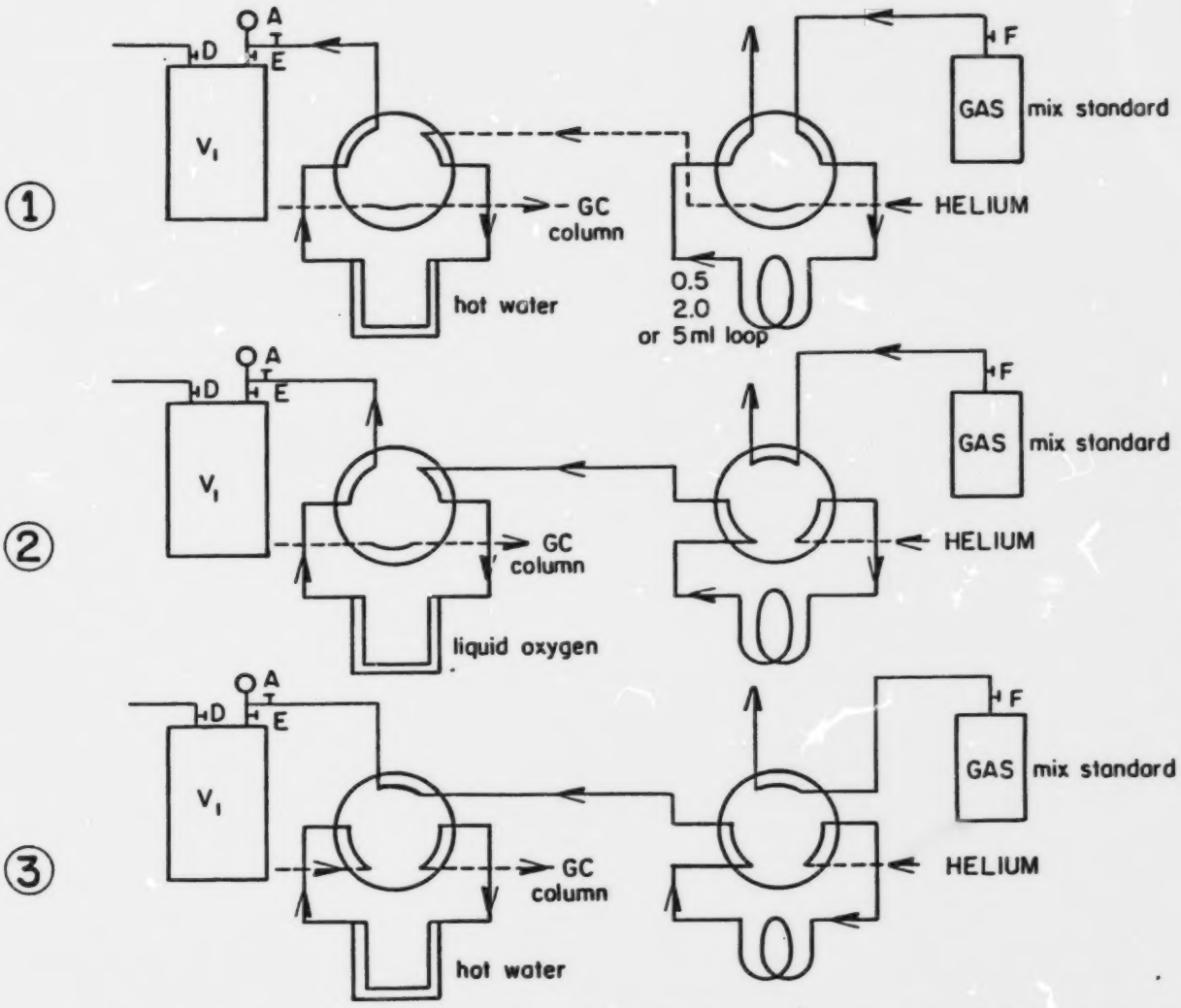


FIGURE 4

Analysis - Setup for Injection of the Sample



1= Flushing of trap, filling of sampling loop (0.5, 2 ml or 5 ml). Valves A, D, E and F open.

2= Transfer of standard sample into the cryogenic trap (using 1 litre of helium). Valves A and E open, D and F closed.

3= Injection into the chromatograph column. Valves A, D and F closed.

Note: If required, phases 1 and 2 can be repeated several times prior to injection (phase 3). In that case phase 1 is repeated with valves A and D closed and liquid oxygen is used instead of hot water, to hold the sample in the trap.

FIGURE 5

Analysis - Setup for Calibration

3.1.3 Injection procedure. The sample injection procedure was divided into three parts: preconcentration, flushing and injection.

1. Preconcentration

The 6-port valve was set to fill position. Valves A, B, C, D and E were all open at first and valve H was closed (see Figure 4). The trap was immersed in boiling water for 30 seconds, and then the analysis proceeded as follows:

- Close valve E; allow the pressure to rise to about 240 KPa (20 psig) on PG2 and quickly close B (caution is necessary, for the pressure increases rapidly; valve B should be open only very slightly).

- Wait a minute to ensure there are no leaks.

- Open valve E and wait 10 seconds.

- Close valve D.

- Note the pressure on PG2 (P_0).

- Replace the boiling water with liquid oxygen, and wait at least 20 seconds after the liquid oxygen has evaporated to ensure that the trap is at the same temperature as the bath.

- Open valve B gradually so that the pressure on PG2 rises very slowly, indicating that the air flow in the trap is very low (too high a flow rate prevents ethane and ethylene from being retained in the trap).

- Close B once the desired sample volume has passed through the trap.

- Note the pressure on PG2 (P_1).

2. Flushing

- Open valve H and flush with a litre of helium, to remove nitrogen and oxygen. The pressure difference on PG2 again indicates the volume of gas which has passed through the trap.

- Close valve A.

- Set the 6-port valve to the injection position (see Figure 4).

3. Injection

- Wait about one minute to allow the pressures to stabilize.

- Quickly replace the liquid oxygen with boiling water and start the GC recorder integrator.

3.1.4 Calibration of the instrument.

Flame ionization detector response

Three air mixtures, containing 2.99, 48 and 95 ppm of propane respectively, were used to establish the response curves of the flame ionization detector. The setup used is shown in Figure 5. Figure 6 shows that the detector's response varies linearly with concentration.

When 0.5 ml and 2 ml of the standard gas were injected directly, almost 100% of the propane was captured in the trap. It can be assumed that for compounds heavier than propane the trap would also be 100% efficient.

During the analyses, the linearity of responses was verified by injecting 5 ml of air containing 2.99 ppm of propane. The operation was restarted, and phases 1 and 2 (Figure 5) were repeated, so as to double the amount of propane caught in the trap; the response of this second test was almost exactly double that of the first, which confirmed the linearity of the FID response.

The value of the slope in Figure 6 was used to calibrate the integrator.

Retention time

Mixtures of hydrocarbons in nitrogen were used to determine the retention times of the different compounds, which were later confirmed by mass spectrometry.

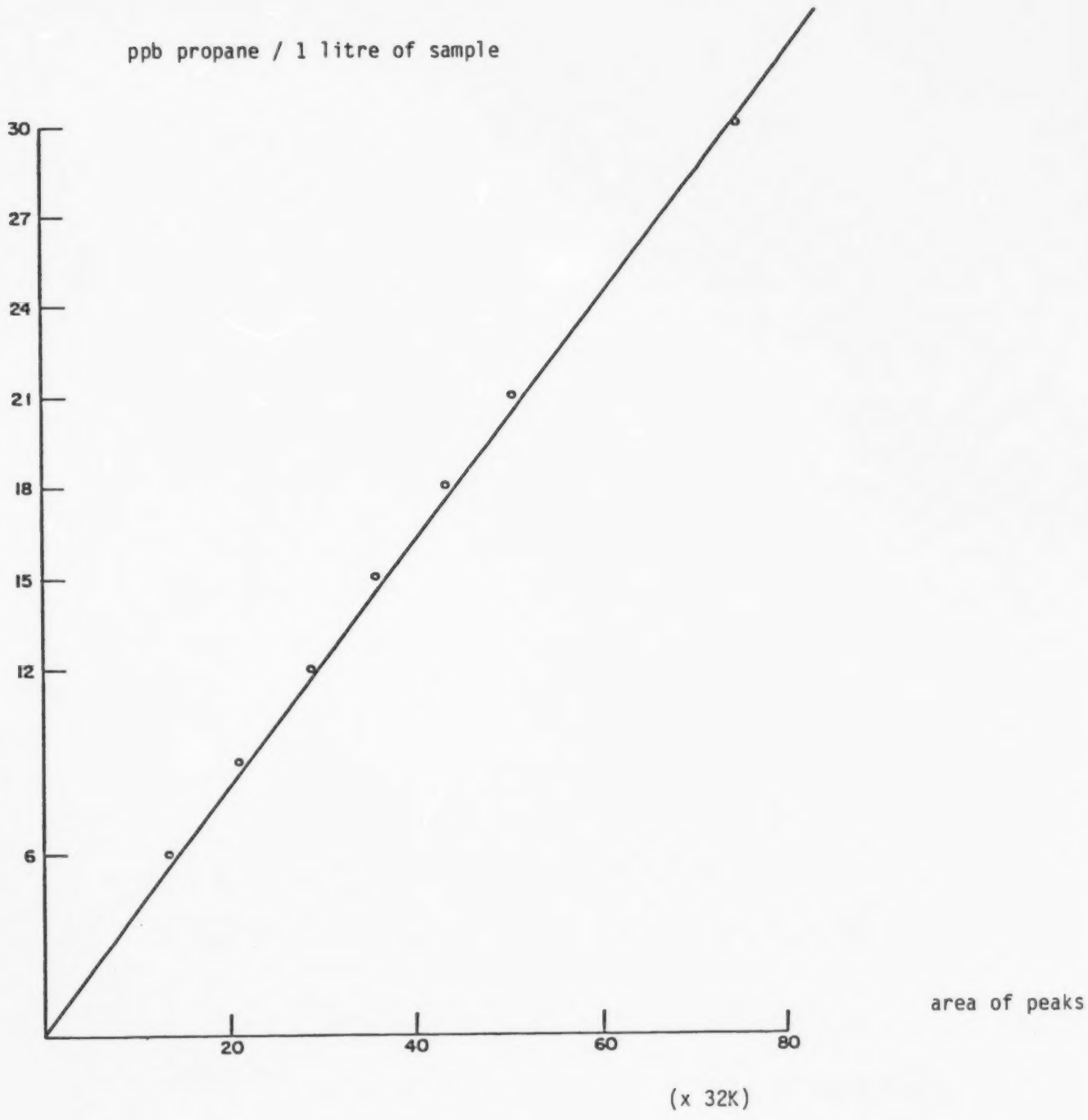


Figure 6

Response of the detector as a function of the concentration of a standard gas

The retention times obtained for each compound are shown below. The concentration was calculated by supposing that the detector's response was directly proportional to the number of carbon atoms.

	Retention Time (1) (minutes)	Concentration measured (ppm)	Concentration recorded on canister (ppm)
	1.43	17.0	17.0
Methane	1.77	10.4	10.3
Ethane	1.81	15.0	15.0
Ethylene	2.51	15.1	20.7
Acetylene	2.59	10.1	10.6
Propane	3.38	13.6	13.4
Propylene	4.19	14.6	14.6
Isobutane	4.73	10.7	10.8
n-Butane	6.93	13.3	13.1
1-Butene			
2,2-Dimethylpropane	6.98	14.5	14.7
2-Methylbutane	8.89	14.0	15.2
n-Pentane	10.14	10.0	10.4
1-Pentene	15.38	14.4	15.2
2,2-Dimethylbutane			
2-Methylpentane)	16.46	14.0	15.4
3-Methylpentane)	20.23	28.3	30.8
Hexane			
Hexene	23.67	9.8	10.1
	37.86	13.1	15.3

(1) See Figure 7

3.2 Results

Each sample was analysed at least twice; the results reported have been confirmed with less than 10% difference, in general. All the results are compiled in Table 1.

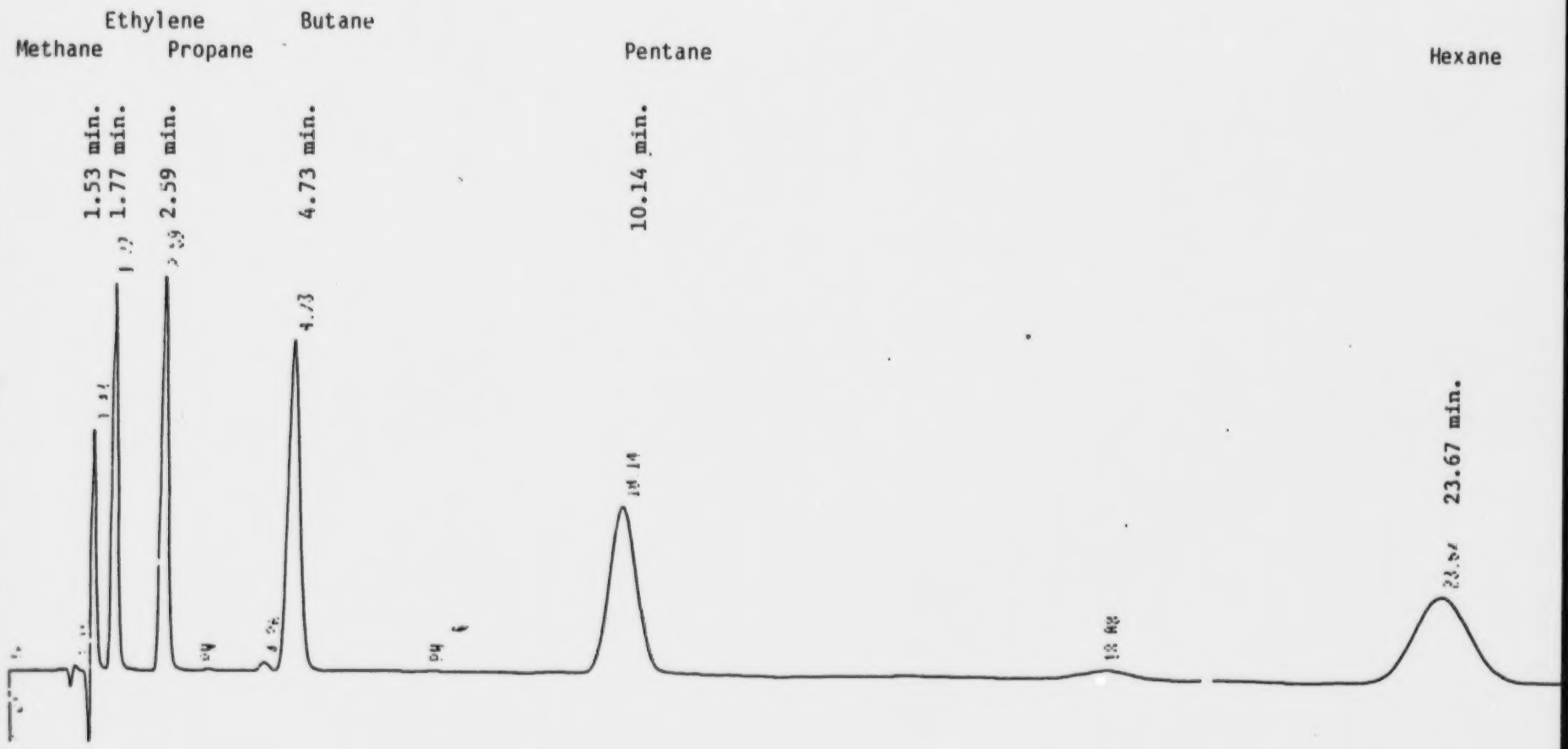


FIGURE 7

Graph obtained by gas chromatography
for the analysis of the standard gas

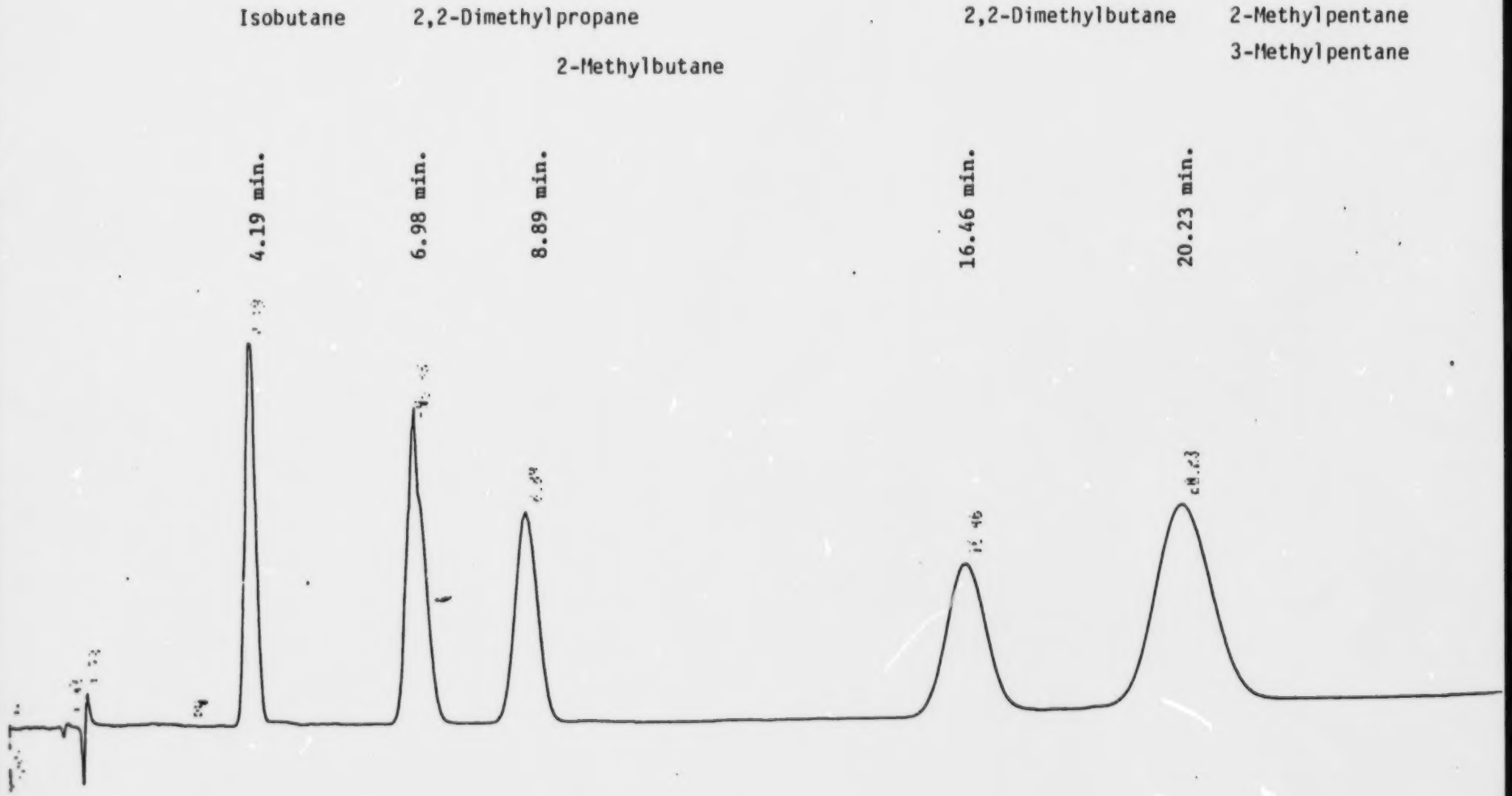


FIGURE 7 (cont'd)

Graph obtained by gas chromatography for the analysis of the standard gas

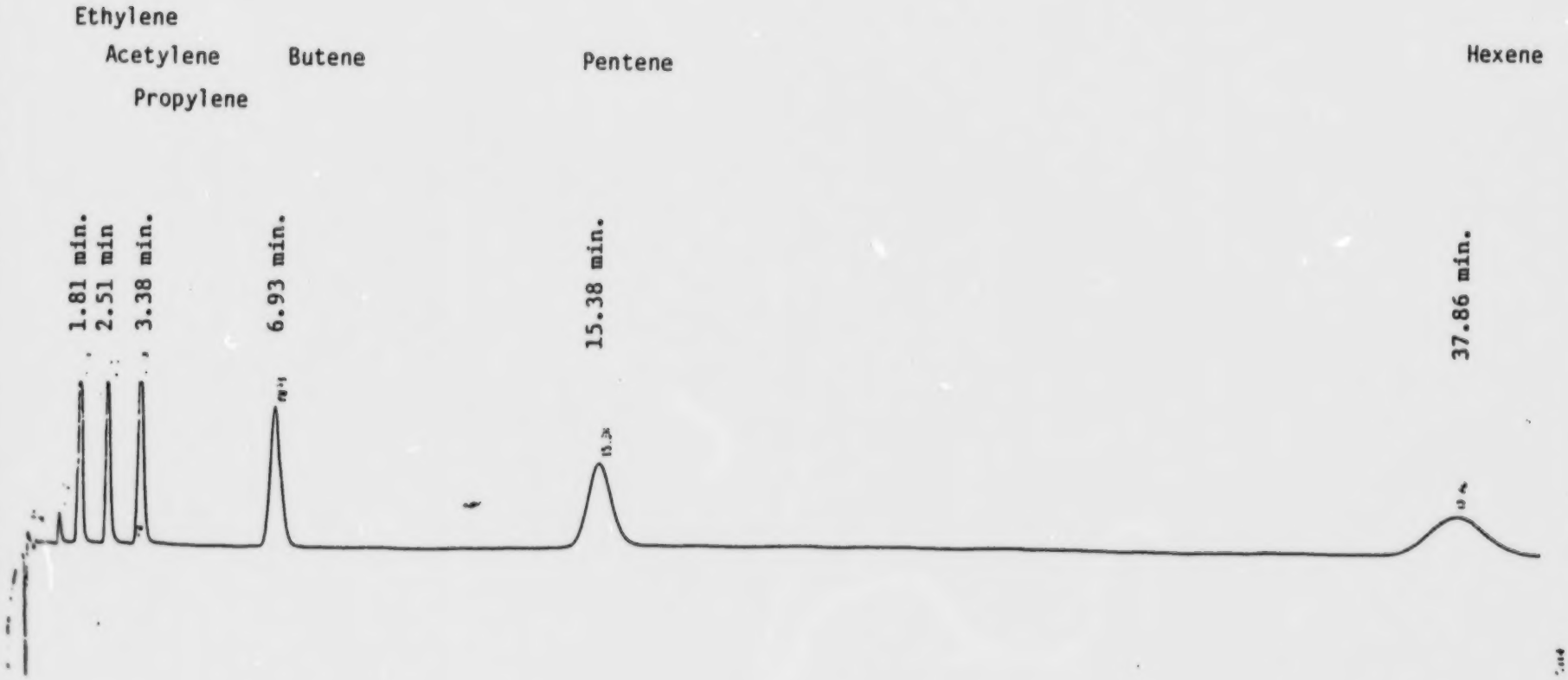


FIGURE 7 (cont'd)

Graph obtained by gas chromatography for the analysis of the standard gas

Table 1

Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site / Date / Time	2 St-Jacques 82-08-31 8 - 11 h	4 St-Jean-Baptiste 82-09-01 7 - 11 h	5 St-Jacques 82-09-01 15 - 18 h	6 St-Jacques 82-09-02 7h40-10h40	7 St-Jacques 82-09-07 15 - 18 h	8 St-Jacques 82-09-07 Blank	9 St-Jacques 82-09-08 7h45-10h45	10 St-Jacques 82-09-08 Blank	12 St-Jean-Baptiste 82-09-08 Blank	13 St-Jean-Baptiste 82-09-10 11h45 - 14h45	14 St-Jacques 82-09-13 8 - 11 h
1.75	Ethane		7.5	—	—	8.4	5.1	N/A	5.4	N/A	N/A	26.4	12.9
1.82	Ethylene		36.6	16.8	54.0	70.2	39.9		54.6			24.0	47.4
2.48	Propane and Acetylene		28.5	11.7	42.9	61.2	17.4		51.3			88.5	44.7
3.38	Propylene (Freon 12)		11.4	9.0	12.9	24.6	9.3		11.1			15.9	14.7
4.16	Isobutane		28.5	2.4	13.2	44.7	21.6		36.9			115.5	22.8
4.63	n-Butane		42.0	3.0	21.9	62.1	34.8		55.2			156.0	33.9
6.98	2,2-Dimethyl propane or Butene		—	3.0	2.7	6.0	6.3		6.6			17.7	5.7
8.21	1-Butene		12.3	0.9	2.7	19.5	13.2		21.9			30.0	12.0
8.87	2-Methyl butane		61.5	7.5	41.1	76.8	47.4		72.9			283.5	60.9
9.96	n-Pentane		30.6	4.5	24.6	54.3	26.7		45.0			151.5	37.2
15.71	2,2-Dimethyl butane		—	—	31.53	10.8	—		—				
	2,3 Dimethyl butane		—									30.9	11.4
19.94	2-Methyl pentane		30.0	—	24.6	47.1	33.3		44.7				37.5
	3-Methyl pentane		—		1.5	12.0	—		—			132.6	
22.67	Hexane		15.0	—	4.5	18.0	14.4		18.6			43.2	13.2
37.86	Hexene		—	—	—	—	—		—			—	
	TOTAL		303.9	58.8	278.1	515.7	269.4	N/A	424.2	N/A	N/A	1115.7	354.3

Table 1 (cont'd)
Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site / Date / Time	15 St-Jacques 82-09-15 15 - 18 h	16 St-Jean-Baptiste 82-09-16 10 h 25 - 13 h 25	17 St-Jean-Baptiste 82-09-17 13 h 30 - 16 h 30	18 St-Jean-Baptiste 82-09-20 12 h 15 - 15 h 15	19 St-Jean-Baptiste 82-09-21 10 h 40 - 13 h 40	20 St-Jacques 82-09-21 15 h 20 18 h 20	21 St-Jacques 82-09-22 15 - 18 h	22 St-Jacques 82-09-23 7 h 30 - 10 h 30	23 St-Jean-Baptiste 82-09-23 12 h 10 - 15 h 10	24 St-Jean-Baptiste 82-09-28 Blank	25 St-Jean-Baptiste 82-09-28 9 h 15 - 12 h 15
1.75	Ethane		—	—	4.2	14.4	—	—	6.9	—	—	Trace(0.3)	9.6
1.82	Ethylene		46.8	143.7	23.7	30.0	32.4	35.1	20.4	62.7	18.5		15.3
2.48	Propane and Acetylene		99.4	159.9	20.4	21.3	21.6	21.0	18.6	48.0	12.3		17.7
3.38	Propylene (Freon 12)		18.6	47.4	6.0	—	3.0	6.0	6.0	—	6.0		6.9
4.16	Isobutane		23.4	151.5	13.5	3.9	28.5	12.3	16.2	26.4	3.0		8.7
4.63	n-Butane		37.2	235.8	26.4	16.2	33.9	18.9	22.5	40.2	4.8		12.3
6.98	2,2-Dimethyl propane or Butene		—	13.8	—	—	—	—		—	—		—
8.21	1-Butene		11.7	—	—	—	—	—	—	—	2.1		2.4
8.87	2-Methyl butane		63.0	274.8	47.7	10.2	31.8	32.1	48.9	59.4	10.2		4.8
9.96	n-Pentane		40.2	140.7	21.6	17.1	19.8	19.8	27.0	39.6	8.1		21.9
15.71	2,2-Dimethyl butane			7.8	—	—	—	—	—	—	—		15.3
	2,3-Dimethyl butane		34.2										—
19.94	2-Methyl pentane		69.0	104.7	22.5	7.5	19.2	29.4	37.2	45.9	15.0		20.7
	3-Methyl pentane		17.1										
22.67	Hexane		12.3	49.2	9.3	—	99.0	14.1	17.7	32.1	5.1		3.6
37.86	Hexene		—	—	—	—	—	—	—	—	—		—
	TOTAL		402.9	1329.3	195.3	120.6	199.2	188.7	221.4	354.3	85.2	Trace(0.3)	139.2

Table 1 (cont'd)
Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site / Date / Time	26 St-Jacques 82-09-28 Blank	27 St-Jacques 82-09-28 15 h 5 - 18 h 5	28 St-Jacques 82-10-04 15 - 18 h	29 St-Jean-Baptiste 82-10-05 9 h 5 - 12 h 5	30 St-Jean-Baptiste 82-10-06 8 h 55 - 11 h 551	31 St-Jacques 82-10-06 14 h 45 - 17 h 45	32 St-Jean-Baptiste 82-10-07 9 h 30 - 12 h 30	33 St-Jean-Baptiste 82-10-14 9 - 12 h	34 St-Jacques 82-10-14 15 - 18 h	35 St-Jacques 82-10-15 7 h 45 - 10 h 45	36 St-Jacques 82-10-19 7 h 40 - 10 h 40
1.75	Ethane		6.0	18.6	11.4	18.3	130.5	31.8	4.5	38.4	38.1	25.2	43.5
1.82	Ethylene		3.6	45.3	30.0	63.9			9.6				
2.48	Propane and Acetylene		2.1	36.9	17.7	85.3	130.5	21.0	6.9	73.5	21.6	15.3	27.6
3.38	Propylene (Freon 12)		2.1	11.4	8.7	18.0	27.0	8.7	4.8	15.6	9.3	5.7	10.5
4.16	Isobutane		2.4	42.9	24.0	78.6	130.5	13.2	2.4	45.9	14.7	8.4	13.5
4.63	n-Butane		4.2	51.6	27.0	139.2	174.3	18.6	4.2	72.6	18.9	10.8	18.6
6.98	2,2-Dimethyl propane or Butene		—	—	--	—	—	—		—	—	—	--
8.21	1-Butene		—	9.6	3.9	16.2	20.4	5.4	4.2 1.8	9.9	7.8	3.9	6.9
8.87	2-Methyl butane		4.5	78.0	52.2	173.4	196.8	31.5	4.2	77.4	34.5	19.5	34.5
9.96	n-Pentane		2.7	42.6	24.0	118.8	110.4	20.4	3.6	43.8	21.9	12.0	22.8
15.71	2,2-Dimethyl butane		—	6.9	7.2	4.2	6.3	1.2	2.7	—	1.8	1.5	--
	2,3-Dimethyl butane			12.6				5.1					
19.94	2-Methyl pentane 3-Methyl pentane		2.4	67.5	45.9	103.5	137.4	24.9	5.7	49.5	28.2	21.0	33.3
22.67	Hexane		--	42.3	28.6	27.3	33.8	9.9	3.3	19.2	18.0	6.9	13.2
37.86	Hexene		--	—	--	—	—	—	—	15.3	—	--	15.0
	TOTAL		30.0	466.2	270.6	847.2	1097.9	191.7	57.9	461.1	214.8	130.2	239.4

Table 1 (cont'd)
Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site	37 St-Jacques	38 St-Jacques	39 St-Jean-Baptiste	40 St-Jean-Baptiste	41 St-Jean-Baptiste	42 St-Jacques	43 St-Jacques	44 St-Jacques	45 St-Jean-Baptiste	46 St-Jean-Baptiste	47 St-Jean-Baptiste
		Date	82-10-20	82-10-21	82-10-22	82-10-26	82-10-27	82-10-28	82-10-28	82-10-29	82-10-29	82-11-03	82-10-03
		Time	15 - 18 h	15 - 18 h	9 - 12 h	15 - 18 h	9 - 12 h	6 h 50 - 9 h 50	15 - 18 h	6 h 45 - 9 h 45	15 h 04 18 h 04	8 h 30 - 11 h 30	15 h 15 18 h 15
1.75	Ethane		33.3	3.6	2.7	6.3	92.4	57.0	14.7	31.5	63.6	10.5	31.5
1.82	Ethylene			22.5	15.0	24.3	253.5		91.5	58.2		82.5	67.5
2.48	Propane and Acetylene		20.4	12.9	7.8	5.7 7.5	253.5	42.0	55.5	43.5	5.7 44.4	54.0	183.9
3.38	Propylene (Freon 12)		6.9	4.8	4.2	6.3	38.7	12.0	18.6	15.3	7.2	17.1	52.8
4.16	Isobutane		11.7	8.1	2.7	5.1	145.8	45.0	76.8	46.2	62.4	37.8	163.5
4.63	n-Butane		16.2	10.2	4.2	7.8	241.8	76.5	112.5	75.9	92.4	44.4	216.9
6.98	2,2-Dimethyl propane or Butene		—	—		0.6	1.5	10.5	14.4	12.0	6.6	0.6	0.9
8.21	1-Butene		3.0	2.1	1.8	3.0	13.2	27.0	37.8	31.5	15.6	4.5	17.7
8.87	2-Methyl butane		24.6	20.1	8.7	15.3	260.7	81.0	122.4	79.5	101.7	68.7	302.4
9.96	n-Pentane		15.0	10.2	6.0	7.2	186.6	33.0	67.8	39.3	68.7	40.2	167.1
15.71	2,2-Dimethyl butane		1.8	1.8	1.2	1.2	6.3	3.0	5.1	4.5	7.2	—	
	2,3-Dimethyl butane					0.3	6.9	7.8	6.9	14.4	5.1		18.9
19.94	2-Methyl pentane 3-Methyl pentane		19.8	17.1	7.5	16.5	222.0	45.0	72.0	86.4	87.9	53.1	219.6
22.67	Hexane		8.4	7.5	4.5	10.2	87.0	18.9	25.2	18.3	33.9	21.0	83.7
37.86	Hexene		—	—	—	—	50.1	—	—	—	—	—	65.1
	TOTAL		161.1	120.9	66.3	117.3	1860.0	458.7	721.2	556.5	602.4	434.4	1591.5

Table 1 (cont'd)
Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site	48 St-Jacques	49 St-Jean-Baptiste	50 St-Jean-Baptiste	51 St-Jacques	52 St-Jacques	53 St-Jean-Baptiste	54 St-Jean-Baptiste	55 St-Jean-Baptiste	56 St-Jean-Baptiste	57 St-Jacques	58 St-Jacques
		Date	82-11-04	82-11-05	82-11-08	82-11-09	82-11-10	82-11-10	82-11-15	82-11-16	82-11-17	82-11-18	82-11-22
		Time	7 - 10 h	8 h 15 - 11 h 15	14 - 17 h	10 h 30 - 13 h 30	6 h 50 - 9 h 50	12 h 5 - 15 h 5	14 h 40 - 17 h 40	14 h 30 - 17 h 30	14 h 05 - 17 h 05	10 h 50 - 13 h 50	15 - 18 h
1.75	Ethane		12.9	—	12.0	8.7	5.4	219.9	14.4	6.3	151.5	22.2	19.8
1.82	Ethylene		97.5	77.4	45.9	30.7	92.7	184.8	43.5	31.2		56.4	56.4
2.48	Propane and Acetylene		75.3	11.1	34.2	17.4	60.6	101.4	75.9	21.0	210.0	49.5	37.0 32.6
3.38	Propylene (Freon 12)		19.8	22.8	9.0	6.3	23.1	12.3	15.9	3.6	24.3	18.0	35.4
4.16	Isobutane		74.4	89.7	29.1	35.4	32.7	230.1	66.0	10.5	120.0	29.3	90.0
4.63	n-Butane		85.2	106.5	39.3	36.6	41.1	267.6	88.8	16.5	264.9	2340.0	107.1
6.98	2,2-Dimethyl propane or Butene		110.2	10.2	0.3	3.6	5.7	12.9		—	—	—	—
8.21	1-Butene		25.2	23.7	2.7	5.1	12.0	29.4	6.1	1.2	5.7	3.9	11.1
8.87	2-Methyl butane		96.3	97.5	46.8	34.8	62.4	221.1	90.0	21.0	167.7	57.3	130.6
9.96	n-Pentane		51.9	55.2	25.2	19.8	39.0	112.8	48.0	13.5	114.3	30.6	63.0
15.71	2,2-Dimethyl butane					0.9	1.5	2.7	3.9	2.7	5.7	6.3	9.3
	2,3-Dimethyl butane		15.9	15.0	4.2	3.0	6.6	11.7					
19.94	2-Methyl pentane 3-Methyl pentane		55.8	57.9	33.9	45.0	71.1	111.6	58.8	21.9	115.8	54.0	84.6
22.67	Hexane		24.0	21.3	12.3	24.9	27.3	42.3	21.3 26.7	9.3	38.0 34.2	27.0 36.0	43.8 51.3
26.04 37.86	Hexene		31.8	—	—	—	35.7	8.7	25.5	—	92.7	30.0	19.2
	TOTAL		676.2	588.3	294.9	272.1	516.9	1569.3	584.8	158.7	1344.8	2760.5	791.2

Table 1 (cont'd)
Analysis results (concentration in ppb C)

Elution time	Compounds	Sampling site / Date / Time	59 St-Jacques 82-11-23 11 - 14 h	60 St-Jean-Baptiste 82-11-24 14 h 45 - 17 h 45	61 St-Jean-Baptiste 82-11-24 Blank	62 St-Jean-Baptiste 82-11-25 8 h 15 - 11 h 15	63 St-Jean-Baptiste 82-11-25 13 h 30 - 16 h 30	64 St-Jean-Baptiste 82-11-30 13 h 10 - 16 h 10	65 St-Jean-Baptiste 82-12-01 8 h 30 - 11 h 30	66 St-Jacques 82-12-01 14 h 40 - 17 h 40	67 St-Jacques 82-12-02 7 h 10 - 10 h 10	68 St-Jean-Baptiste 82-12-02 13 h 25 - 16 h 25
1.75	Ethane		16.2	42.3	22.8	72.0	12.1	59.4	78.4	255.0	124.8	103.5
1.82	Ethylene		61.2		6.9	78.0	28.5					
2.48	Propane and Acetylene		60.1	46.2	6.1	261.0	32.7	18.9	28.6	195.0	114.4	63.6
3.38	Propylene (Freon 12)		28.8	8.4		73.8	5.7	17.4	16.2	74.7	43.8	31.9
4.16	Isobutane		67.2	52.3	N/A	110.7	48.9	8.1	8.2	154.1	72.0	34.8
4.63	n-Butane		81.6	62.1	N/A	252.2	90.4	12.4	15.4	217.4	112.1	52.1
6.98	2,2-Dimethyl propane or Butene		—	—		—	—	—			—	
8.21	1-Butene		21.0	5.7	N/A	13.8	0.7	1.2	1.5 1.2	0.6 18.3	11.1	3.6 5.7
8.87	2-Methyl butane		101.4	75.0	N/A	196.8	96.1	20.7	22.5	274.3	171.2	72.0
9.96	n-Pentane		45.3	40.8	N/A	95.4	85.2	11.1	13.9	128.1	90.1	40.8
15.71	2,2-Dimethyl butane 2,3-Dimethyl butane		4.2	8.7	N/A	6.9	2.7	1.8	2.3	17.1	13.2	3.0
19.94	2-Methyl pentane 3-Methyl pentane		55.2	62.2	N/A	165.0	56.0	22.8	24.3	187.2	151.3	61.7
22.67	Hexane		24.6	27.3	N/A	36.0	28.0	12.0	11.6	86.1	71.3	
26.04										30.0	24.4	24.9
37.86	Hexene		—	—		—	—	—	—	120.1	90.0	—
	TOTAL		566.8	431.0	35.8	1361.6	487.0	185.8	224.1	1758.2	1089.7	497.6

3.3 Mass spectrometry analysis

3.3.1 Equipment and methodology. Four samples were analysed by mass spectrometry in order to determine the composition of the various compounds as precisely as possible. The 6-metre (20 foot) column type Durapack Phenyl Isocyanate/Porasil C, 80/100 mesh, which was used for all the chromatography analyses, was first used to analyse samples 27 and 30. The samples were enriched by means of a jet separator. The column was connected to a Finnigan 3200 mass spectrometer detector, and maintained at a constant 45°C. The mass spectrometer was operated in the electron impact mode and scanned between m/e 10 and 200. The data were stored and processed using a Finnigan Incos Data System.

A 30-metre capillary column type SE54 fused silica (J and W) was next used for samples 62 and 66, connected directly to the ion source via a glass-lined tubing interface (so as to eliminate the jet separator).

The temperature of the column was roughly programmed with dry ice between -50°C and the ambient temperature. The spectra were taken at a rate of one scan per second between masses 10 to 400.

3.3.2 Results. The results of the first series of analyses (samples 27 and 30) are shown in Appendix 1, and those of the second series, when the capillary column was used (samples 62 and 66), in Appendix 2.

In the mass spectrometry study, it was not possible to obtain the information desired on some light hydrocarbons, that is ethane, ethylene and acetylene, because:

1. During the first series of analyses the light gases were partially eliminated with the helium as they passed through the separator. The sample was also contaminated by water and carbon dioxide, making readings either impossible owing to mass interference due to nitrogen, or difficult owing to the weakness of the signals detected as compared with the interference.

2. During the second series of analyses (with a capillary column), readings could not be obtained for C_2 and C_3 owing to interference caused by air, water and carbon dioxide (CO_2) in the same zone of detection as C_2 and C_3.

For gases heavier than propane, readings were obtained in all cases.

The mass spectrometry analysis for samples 27 and 30 confirmed the identification of each of the products obtained by chromatography (flame ionization detector [FID]) and that some peaks were mixed. The 2-methylpentane peak actually also contains 3-methylpentane. These two compounds were separated by the capillary column for samples 62 and 66; it seems that 2-methylpentane was the more plentiful (two to three times more). Mass spectrometry analyses revealed the presence of a number of butenes are shown in the table of results at elution times 6.98 and 8.21.

Chlorinated products, such as dichloromethane, 1,1,1-Trichloroethane, carbon tetrachloride, tetrachlorethylene and freon (freon 113 or freon TF) were detected. Their concentration was roughly evaluated at between 10 and 20 ppb C (by comparing their mass spectra).

An analysis using the 15-metre capillary column type SE30 fused silica (J and W), with a flame ionization detector (the oven temperature was -20°C for 10 minutes, and then progressively raised to the ambient temperature), permitted the following comparison for sample 66:

	Capillary column (ppb C)	Isocyanate column (ppb C)
Ethane, ethylene, acetylene	66	255
Propane, propylene	252	269
Isobutane	153	154
Butane	267	217
2-methylbutane	180	274
Pentane	136	128
2-methylpentane, 3-methylpentane	92	187
Hexane	60	86
Toluene	165	-

In addition, the chromatogram revealed 22 non-identified compounds from C_5 to C_7, for a total of 380 ppb C. Seven of these compounds appeared in appreciable quantities (about 20 to 40 ppb C). If these results are compared with the mass spectra obtained with the capillary column, the compounds could correspond to the products identified as hexene, methylcyclopentane, cyclohexane, 2,4-dimethylpentane and n-heptane. This shows that the isocyanate column provides only limited data on C_6 compounds and is really efficient only for compounds C_2 to C_5.

3.4 Comments on the method

- During the gas chromatography it proved necessary to flush with helium so as to rid the trap of air. This made it possible to obtain chromatograms with no disturbances in the initial part of the curve.

- Different tests were conducted so as to check the efficiency of the cryogenic trap with regard to ethane and ethylene.

Air or helium containing these compounds was passed through the trap once or twice, and revealed that the ethane and ethylene were only partially trapped. These results were confirmed in an article in reference 2, where it is recommended that the trap be lengthened to 15 cm (6 inches) in order to increase its efficiency.

During these analyses it was noted that the results obtained for ethane and ethylene fluctuated; the highest concentrations obtained were reported.

- The use of boiling water during the injection of the sample was not a critical factor; lower temperatures can be used without affecting the results.

3.5 Quality control

Ten of the samples taken were transferred into two separate canisters, so that one of each could be sent to the Environment Canada River Road Laboratory, in Ottawa, for analysis.

They were analysed in the same way as the samples analysed by the contractor.

Table 2 compares the results obtained by the contractor with those obtained by the departmental laboratory. In general, there were differences of less than 10% (except perhaps for ethane and ethylene, because of the inefficiency of the freezeout loop where these substances are concerned, as discussed above), which is considered fully satisfactory.

Table 2
Quality control

Compounds	Sample 13		Sample 14		Sample 25		Sample 27		Sample 38	
	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor
Ethane	39.9	26.4	—	12.9	12.1	9.6	34.6	18.6	9.1	3.6
Ethylene		24.0	--	47.4	13.47	15.3	33.8	45.3		22.5
Propane	81.9	88.5	45.0	44.7	29.8	17.7	42.3	36.9	15.5	12.9
Propylene	16.8	15.9	16.9	14.7	7.3	6.9	22.9	11.4	5.8	4.8
Isobutane	118.8	115.5	25.6	22.8	7.5	8.7	51.6	42.9	7.8	8.1
n-Butane	178.8	156.0	34.9	33.9	10.4	12.3	63.1	51.6	10.7	10.2
2,2-Dimethyl propane	16.2	17.7	16.4	5.7	9.8	2.4	7.6	—		—
1-Butene	28.8	30.0	15.1	12.0	8.8	4.8	14.8	9.6	3.0	2.1
2-Methyl butane	266.7	283.5	67.8	60.9	22.8	21.9	105.1	78.0	19.9	20.1
n-Pentane	146.1	151.5	44.8	37.2	12.5	15.3	52.1	42.6	10.6	10.2
2,3-Dimethyl butane		30.9		11.4		—	94.8	19.5	—	1.8
2-Methyl pentane	178.2		41.8		18.8					
+3-Methyl pentane		132.6		37.5		20.7		67.5	17.9	17.1
Hexane	95.7	43.2	20.2	13.2	7.8	3.6	26.8	42.3	6.6	7.5
Hexene	—	—								
TOTAL	1167.4	1115.7	328.5	354.3	161.1	139.2	549.5	466.2	106.9	120.9

Table 2 (cont'd)
Quality control

Compounds	Sample 39		Sample 47		Sample 48		Sample 56		Sample 57	
	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor	RR Lab	Contractor
Ethane	4.1	2.7	28.6	31.5	23.7	12.9	10.2	151.5	19.8	22.2
Ethylene		15.0	94.1	67.5	36.0	97.5	34.8		21.0	56.4
Propane	11.8	7.8	213.3	183.9	71.5	75.3	192.3	210.0	43.5	49.5
Propylene	2.8	4.2	45.9	52.8	19.1	19.8	23.7	24.3	15.0	18.0
Isobutane	2.5	2.7	161.2	163.5	71.4	74.4	120.6	120.0	27.9	29.3
n-Butane	3.7	4.2	203.7	216.9	79.9	85.2	238.8	264.9	2369.4	2340.0
2,2-Dimethyl propane	—	—	—	0.9	95.6	110.2	—	—		—
1-Butene	1.0	1.8	19.7	17.7	9.81	25.2	5.7	5.7	4.2	3.9
2-Methyl butane	1.6	8.7	295.3	302.4		96.3	154.2	167.7	49.5	57.3
n-Pentane	8.2	6.0	168.4	167.1	44.2	51.9	108.3	114.3	26.1	30.6
2,3-Dimethyl butane	5.0	1.2	12.5	18.9	—	15.9	5.7	5.7	3.3	6.3
2-Methyl pentane	8.4	7.5	195.0	219.6	82.5	55.8	79.2	115.8	36.9	54.0
+3-Methyl pentane										
Hexane	4.7	4.5	68.9	83.7	26.7	24	33.6	38.0	21.9	27.0
Hexene										
TOTAL	53.8	66.3	1338.2	1526.4	560.4	644.4	1007.1	1217.9	2638.5	2689.5

CONCLUSION

In general, the sampling method described in this report was strictly followed, so as to avoid any contamination.

The analysis of the samples conducted by the contractor was fully satisfactory, as can be seen if the contractor's results are compared with those of the departmental laboratory.

An examination of the results shows that:

- the average concentration of light hydrocarbons (C_2-C_6) measured at the St. Jacques site was 500 ppb C.

- the average concentration of light hydrocarbons (C_2-C_6) measured at the St. Jean Baptiste site was 600 ppb C.

- concentrations of light hydrocarbons measured at the St. Jacques site do not appear to have been affected by the wind direction.

- at the St. Jean Baptiste site, concentrations of hydrocarbons were generally higher (about twice as much) when the wind was blowing from the northeast (that is, when the wind carried emissions from the oil refineries to the site).

APPENDIX 1

Meteorological data

APPENDIX 1

Weather conditions at time of sampling

Sample number	Date	Site	Wind Direction	Wind Speed (km/hr)	Cloud Cover	Temp (°C)	Total HC	Comments
1	82-08-25	St. Jacques	--	--	--	--	--	Preliminary sample
2	82-08-31	St. Jacques	WSW	3	5/10	16	303.9	
3	82-08-31	St. Jean-Baptiste	NW	15	4/10	--	--	Insuff. pressure
4	82-09-01	St. Jean-Baptiste	NE	10	8/10	12	58.8	for analysis
5	82-09-01	St. Jacques	E	13	7/10	20	278.1	
6	82-09-02	St. Jacques	N	9	10/10	--	515.7	Light rain
7	82-09-07	St. Jacques	E	7	3/10	16	269.4	
8	82-09-07	St. Jacques	--	--	--	--	N/A	Blank
9	82-09-08	St. Jacques	NNE	5	1/10	11	424.2	
10	82-09-08	St. Jacques	--	--	--	--	N/A	Blank
11	82-09-08	St. Jean-Baptiste	SW	9	0/10	18	--	Insuff. pressure for analysis
12	82-09-08	St. Jean-Baptiste	--	--	--	--	N/A	Blank
13	82-09-10	St. Jean-Baptiste	SW	17	4/10	24	1115.7	
14	82-09-13	St. Jacques	-	Calm	0/10	19	354.3	Fog
15	82-09-15	St. Jacques	NE	15	10/10	14	402.9	After rain
16	82-09-16	St. Jean-Baptiste	W	12	10/10	14	1329.3	
17	82-09-17	St. Jean-Baptiste	NW	13	8/10	11	195.3	
18	82-09-20	St. Jean-Baptiste	SE	16	3/10	18	120.6	
19	82-09-21	St. Jean-Baptiste	Variable	2	1/10	16	199.2	
20	82-09-21	St. Jacques	Variable	2	3/10	18	188.7	
21	82-09-22	St. Jacques	E	15	8/10	18	221.4	
22	82-09-23	St. Jacques	NE	8	8/10	11	354.3	
23	82-09-23	St. Jean-Baptiste	NE	8	10/10	13	85.2	Rain
24	82-09-28	St. Jean-Baptiste	--	--	--	--	Trace (0.3)	Blank
25	82-09-28	St. Jean-Baptiste	NW	3	10/10	14	139.2	Light rain
26	82-09-28	St. Jacques	--	--	--	--	30.0	Blank
27	82-09-28	St. Jacques	NE	6	4/10	16	466.2	
28	82-10-04	St. Jacques	W	4	5/10	15	270.6	
29	82-10-05	St. Jean-Baptiste	Variable	2	3/10	11	847.2	
30	82-10-06	St. Jean-Baptiste	SW	7	2/10	14	1097.9	
31	82-10-06	St. Jacques	SW	10	2/10	20	191.7	
32	82-10-07	St. Jean-Baptiste	NE	13	10/10	10	57.9	
33	82-10-14	St. Jean-Baptiste	SW	9	9/10	11	461.1	

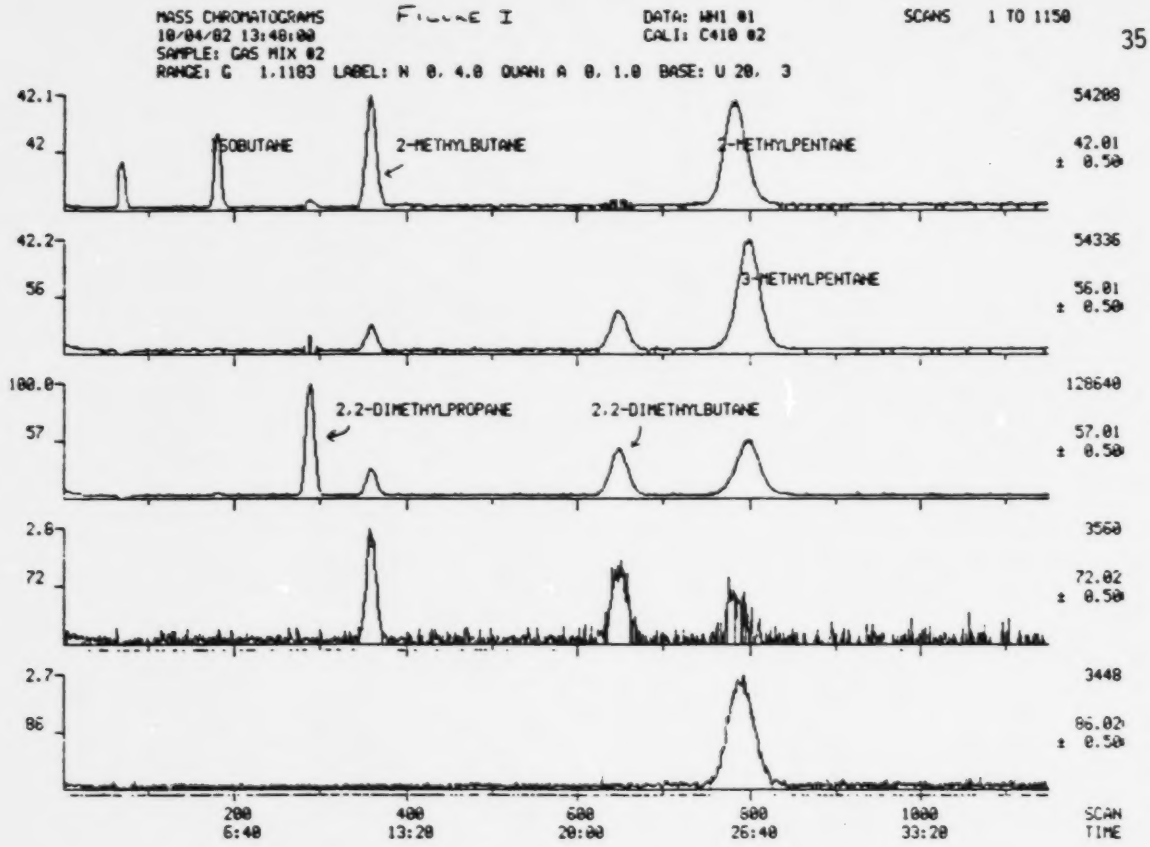
APPENDIX 1

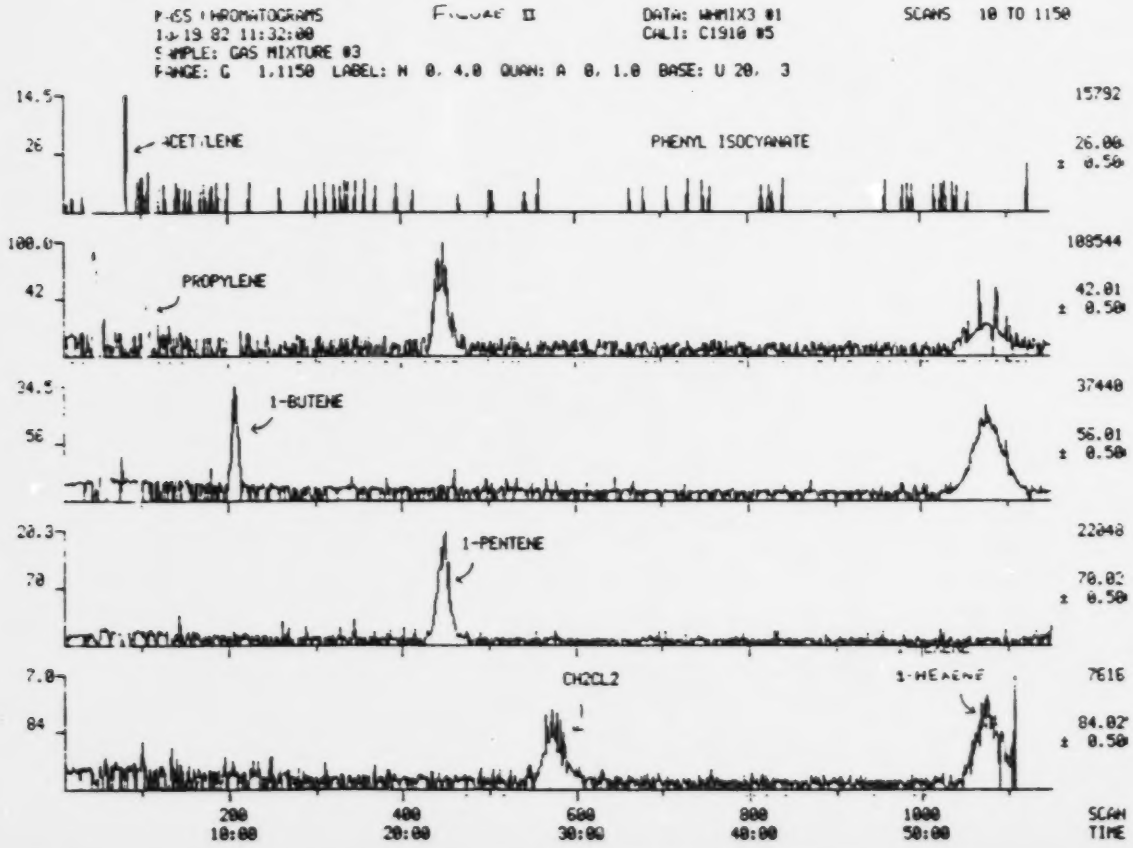
Weather conditions at time of sampling

Sample number	Date	Site	Wind Direction	Wind Speed (km/hr)	Cloud Cover	Temp (°C)	Total HC	Comments
34	82-10-14	St. Jacques	W	6	8/10	12	214.8	Light Rain
35	82-10-15	St. Jacques	SW	15	6/10	9	130.2	
36	82-10-19	St. Jacques	SE	7	5/10	7	239.4	
37	82-10-20	St. Jacques	SE	22	9/10	19	161.1	
38	82-10-21	St. Jacques	W	35	7/10	7	120.9	
39	82-10-22	St. Jean-Baptiste	NW	10	4/10	4	66.3	
40	82-10-26	St. Jean-Baptiste	NE	8	0/10	12	117.3	
41	82-10-27	St. Jean-Baptiste	S	3	5/10	6	1860.0	Foggy
42	82-10-28	St. Jacques	SW	11	1/10	5	458.7	
43	82-10-28	St. Jacques	SW	9	8/10	13	721.2	
44	82-10-29	St. Jacques	SE	6	6/10	10	556.5	
45	82-10-29	St. Jean-Baptiste	SE	11	8/10	17	602.4	
46	82-11-03	St. Jean-Baptiste	--	Calm	10/10	9	434.4	
47	82-11-03	St. Jean-Baptiste	--	Calm	10/10	13	1591.5	Rain
48	82-11-04	St. Jacques	N	15	10/10	12	676.2	Rain
49	82-11-05	St. Jean-Baptiste	SW	33	10/10	5	588.3	
50	82-11-08	St. Jean-Baptiste	NW	14	6/10	14	294.9	
51	82-11-09	St. Jacques	W	20	6/10	5	272.1	
52	82-11-10	St. Jacques	NW	10	0/10	1	516.9	MUCTC strike
53	82-11-10	St. Jean-Baptiste	W	15	9/10	4	1569.3	heavy traffic
54	82-11-15	St. Jean-Baptiste	W	25	10/10	0	584.8	
55	82-11-16	St. Jean-Baptiste	SW	13	4/10	4	158.7	
56	82-11-17	St. Jean-Baptiste	W	9	2/10	6	1344.8	
57	82-11-18	St. Jacques	NW	11	4/10	6	2760.5	
58	82-11-22	St. Jacques	NW	12	10/10	7	791.2	
59	82-11-23	St. Jacques	--	Calm	10/10	9	566.8	Fog
60	82-11-24	St. Jean-Baptiste	W	28	10/10	2	431.0	
61	82-11-24	St. Jean-Baptiste	--	--	--	--	35.8	Blank
62	82-11-25	St. Jean-Baptiste	SW	39	7/10	1	1361.6	
63	82-11-25	St. Jean-Baptiste	W	14	9/10	0	487.0	
64	82-11-30	St. Jean-Baptiste	--	Calm	10/10	1	185.8	Fog
65	82-12-01	St. Jean-Baptiste	N	11	10/10	1	224.1	Mist-fog
66	82-12-01	St. Jacques	N	4	10/10	2	1758.2	
67	82-12-02	St. Jacques	--	Calm	10/10	5	1089.7	
68	82-12-02	St. Jean-Baptiste	NE	3	10/10	8	497.6	

APPENDIX 2

Results of analysis by
mass spectrometry
(Isocyanate column)


MASS CHROMATOGRAMS
10/04/82 13:48:00
SAMPLE: GAS MIX #2
RANGE: G 1.1183 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
DATA: WN1 01
CALI: C410 02
SCANS 1 TO 1150
ISOBUTANE
2-METHYLBUTANE
2-METHYLPENTANE
3-METHYLPENTANE
2,2-DIMETHYLPROPANE
2,2-DIMETHYLBUTANE
SCAN TIME
200 6:40
400 13:20
600 20:00
800 26:40
1000 33:20


SCANS 10 TO 1150
ACETYLENE
PHENYL ISOCYANATE
PROPYLENE
1-BUTENE
1-PENTENE
CH2CL2
1-HEXENE
SCAN TIME
200 10:00
400 20:00
600 30:00
800 40:00
1000 50:00

MASS CHROMATOGRAMS
10/04/82 15:36:00
SAMPLE: HC27
RANGE: G 1,1500 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

FIGURE III

DATA: WHH27 #1
CALI: C410 #2

SCANS 10 TO 1500

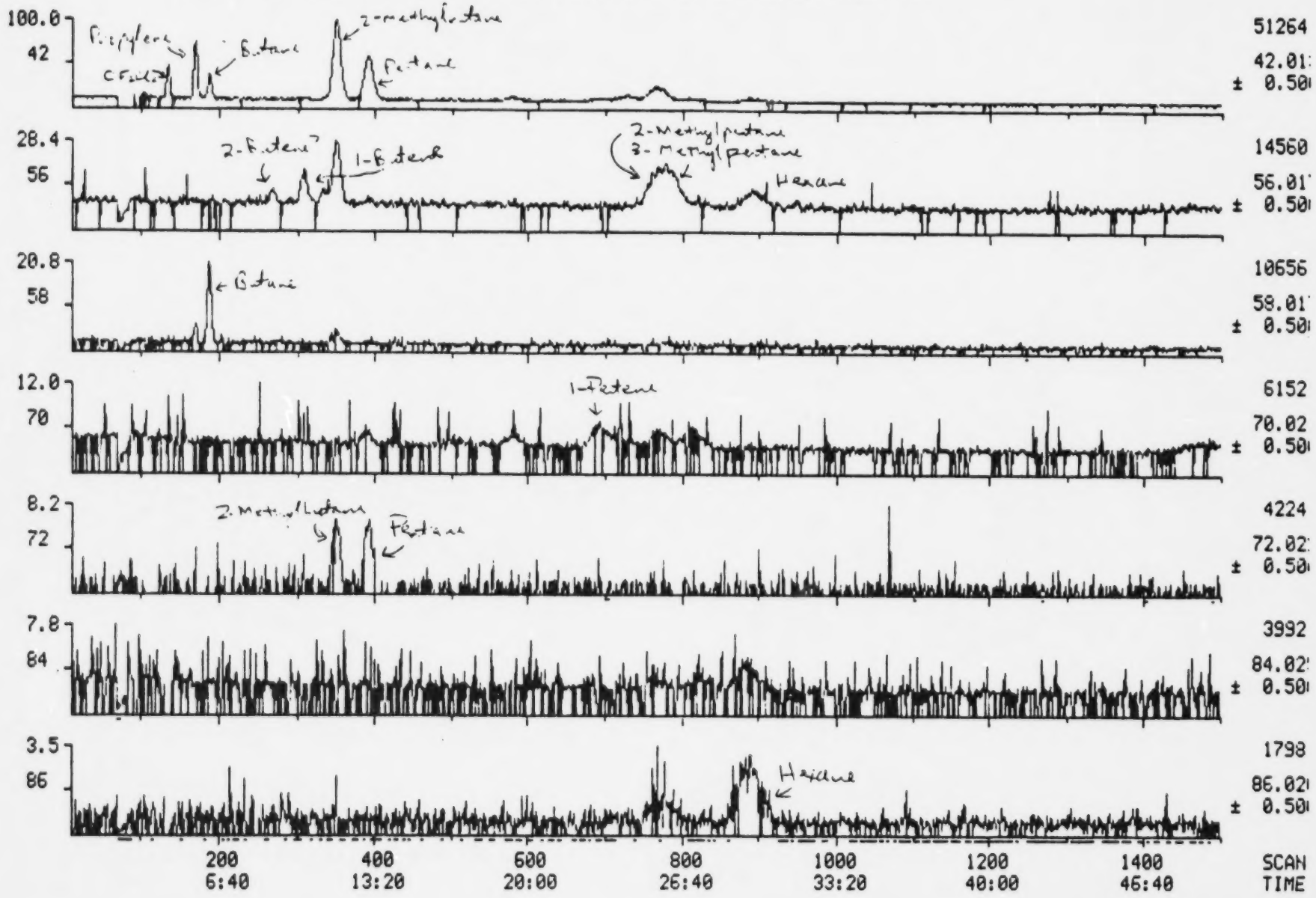
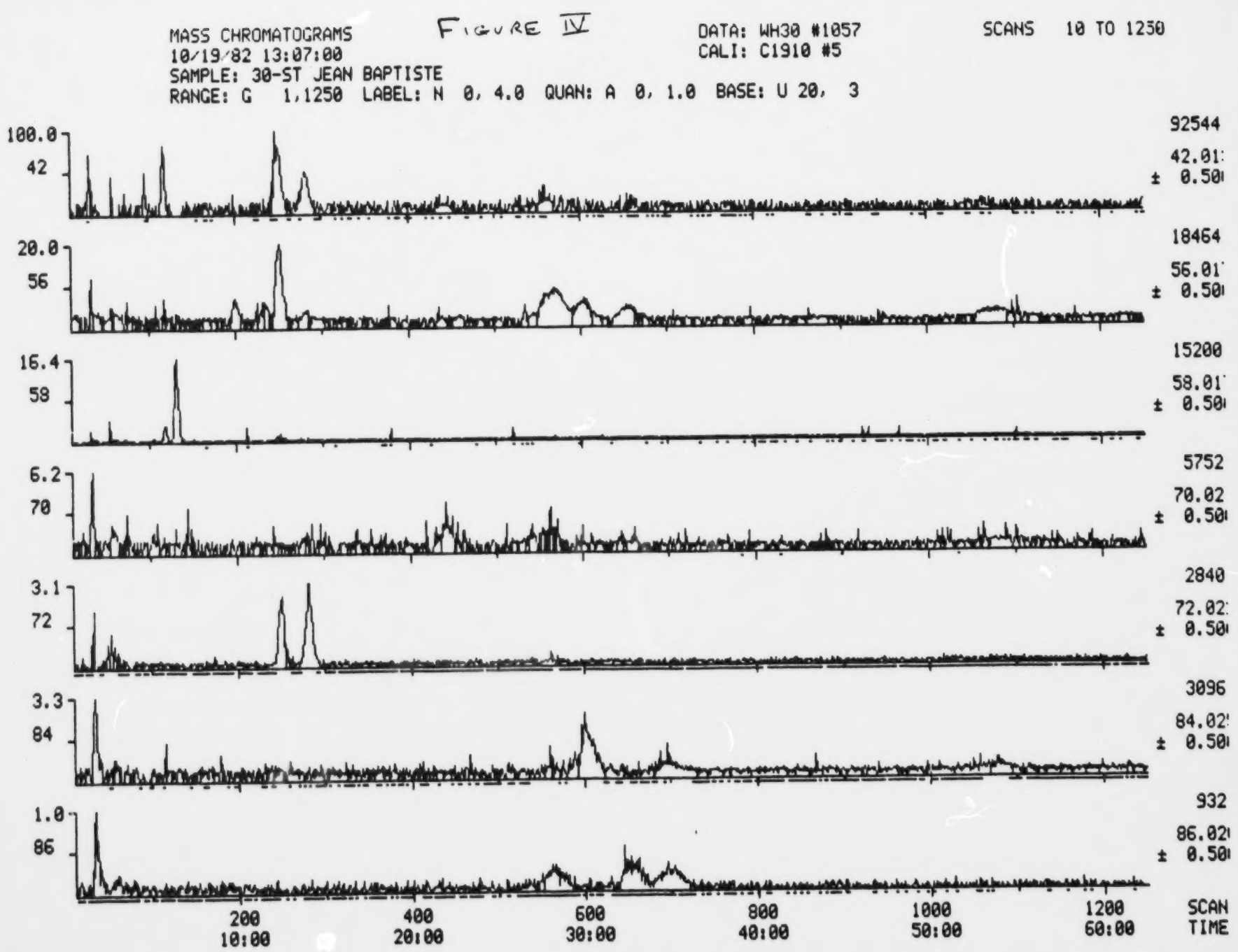


FIGURE IV


MASS CHROMATOGRAMS
10/19/82 13:07:00
SAMPLE: 30-ST JEAN BAPTISTE
RANGE: G 1,1250 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3
DATA: WH30 #1057
CALI: C1910 #5
SCANS 10 TO 1250
100.0
42
92544
42.01
± 0.50
20.0
56
18464
56.01
± 0.50
16.4
58
15200
58.01
± 0.50
6.2
70
5752
70.02
± 0.50
3.1
72
2840
72.02
± 0.50
3.3
84
3096
84.02
± 0.50
1.0
86
932
86.02
± 0.50
200
10:00
400
20:00
600
30:00
800
40:00
1000
50:00
1200
60:00
SCAN
TIME

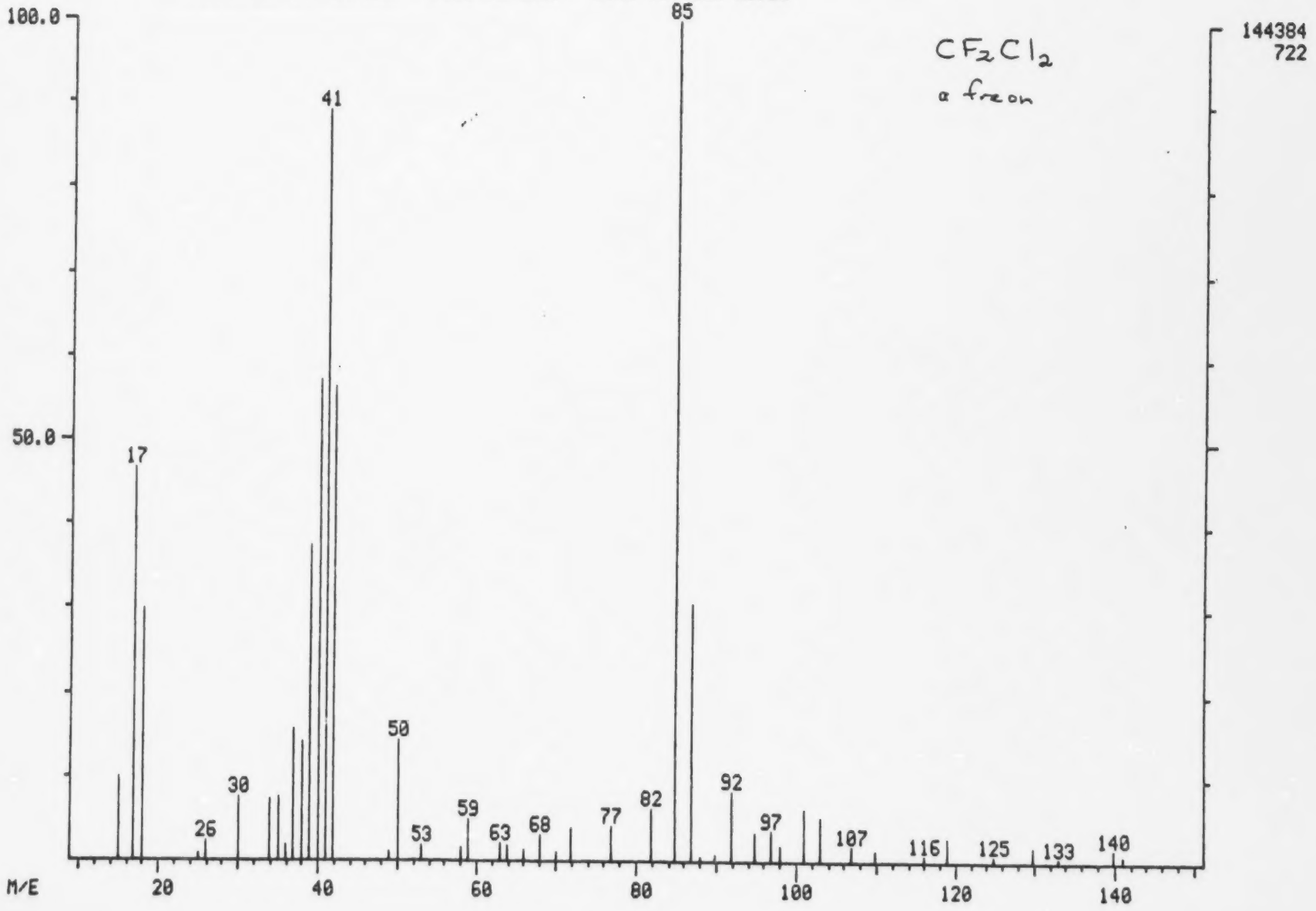
APPENDIX 3

Results of analysis by
mass spectrometry
(capillary column)

MASS SPECTRUM
10/04/82 15:36:00 + 4:32
SAMPLE: HC27
#126 TO #147 SUMMED - #108 TO #121 - #145 TO #156 X1.00

DATA: WHH27 #136
CALI: C410 #2

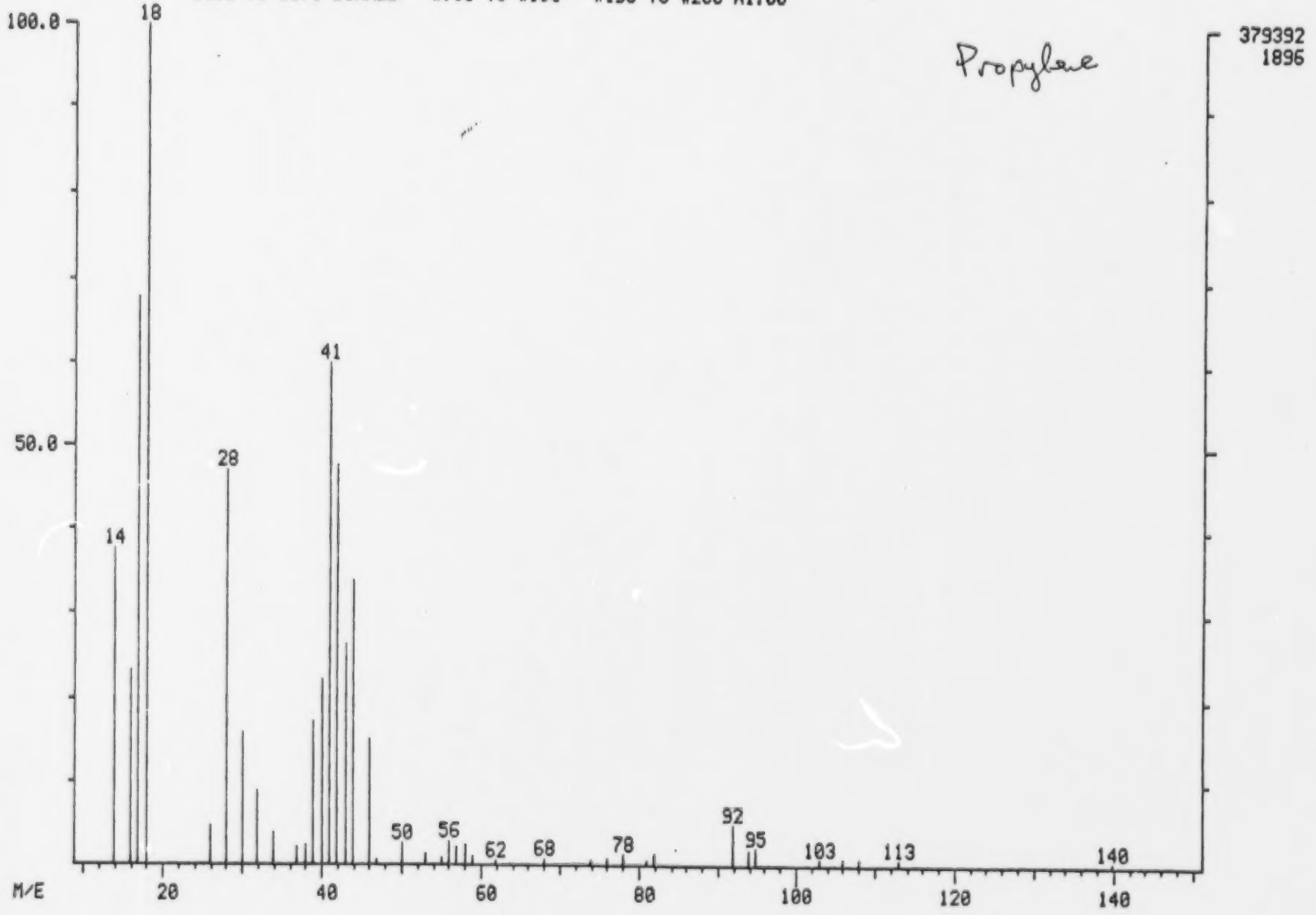
BASE M/E: 85
RIC: 880640.

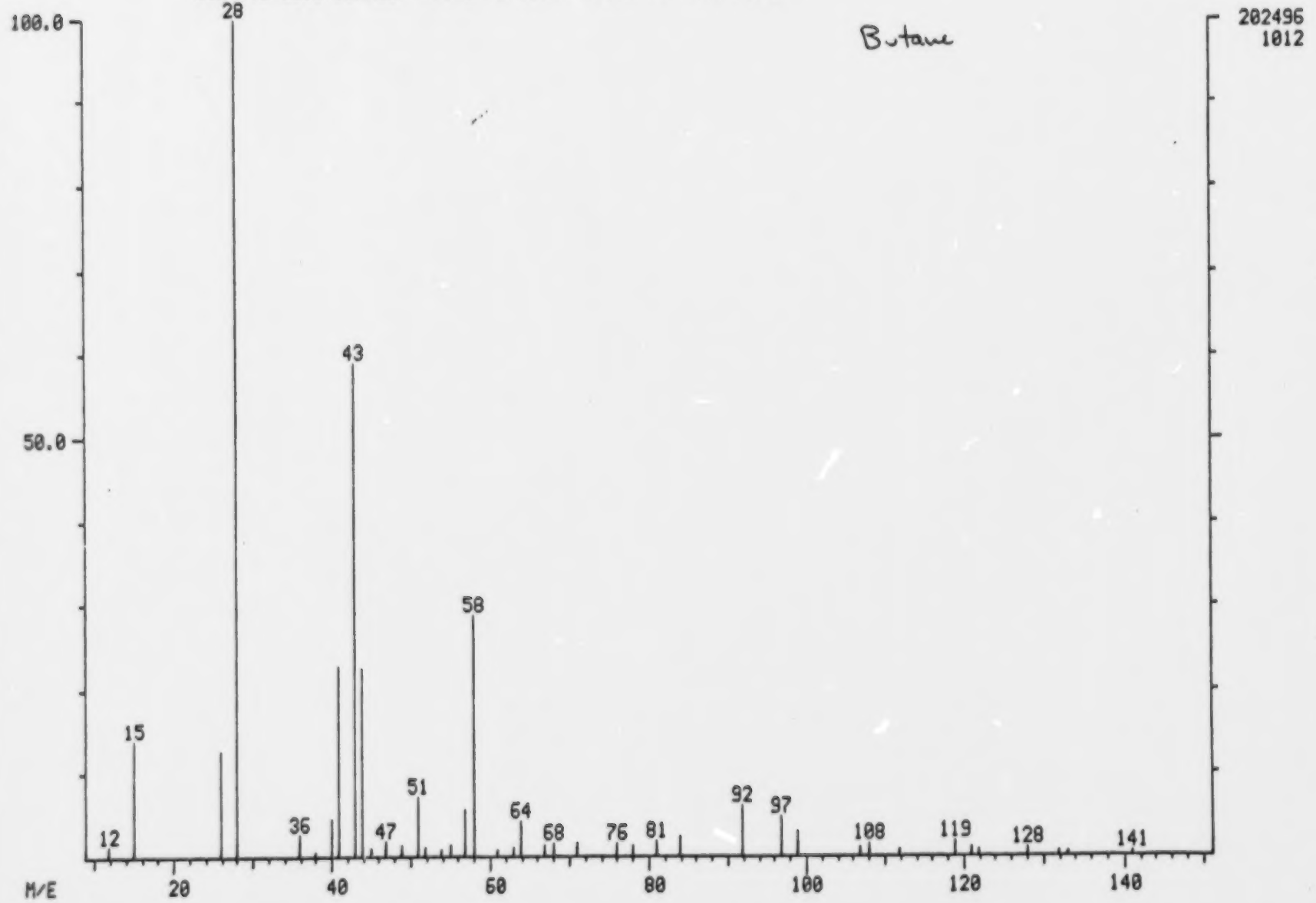


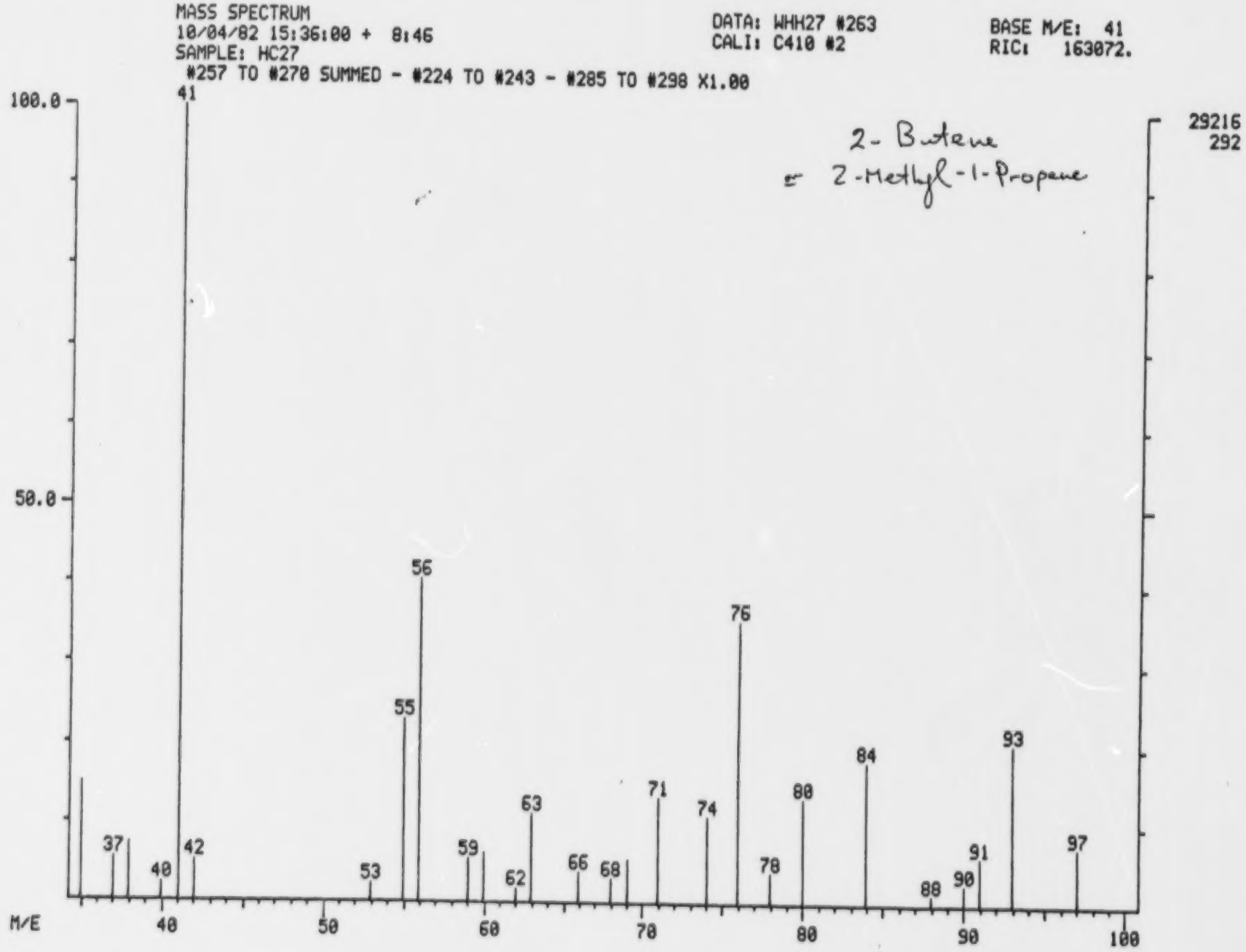
MASS SPECTRUM
10/04/82 15:36:00 + 5:34
SAMPLE: HC27
#158 TO #176 SUMMED - #150 TO #153 - #196 TO #200 X1.00

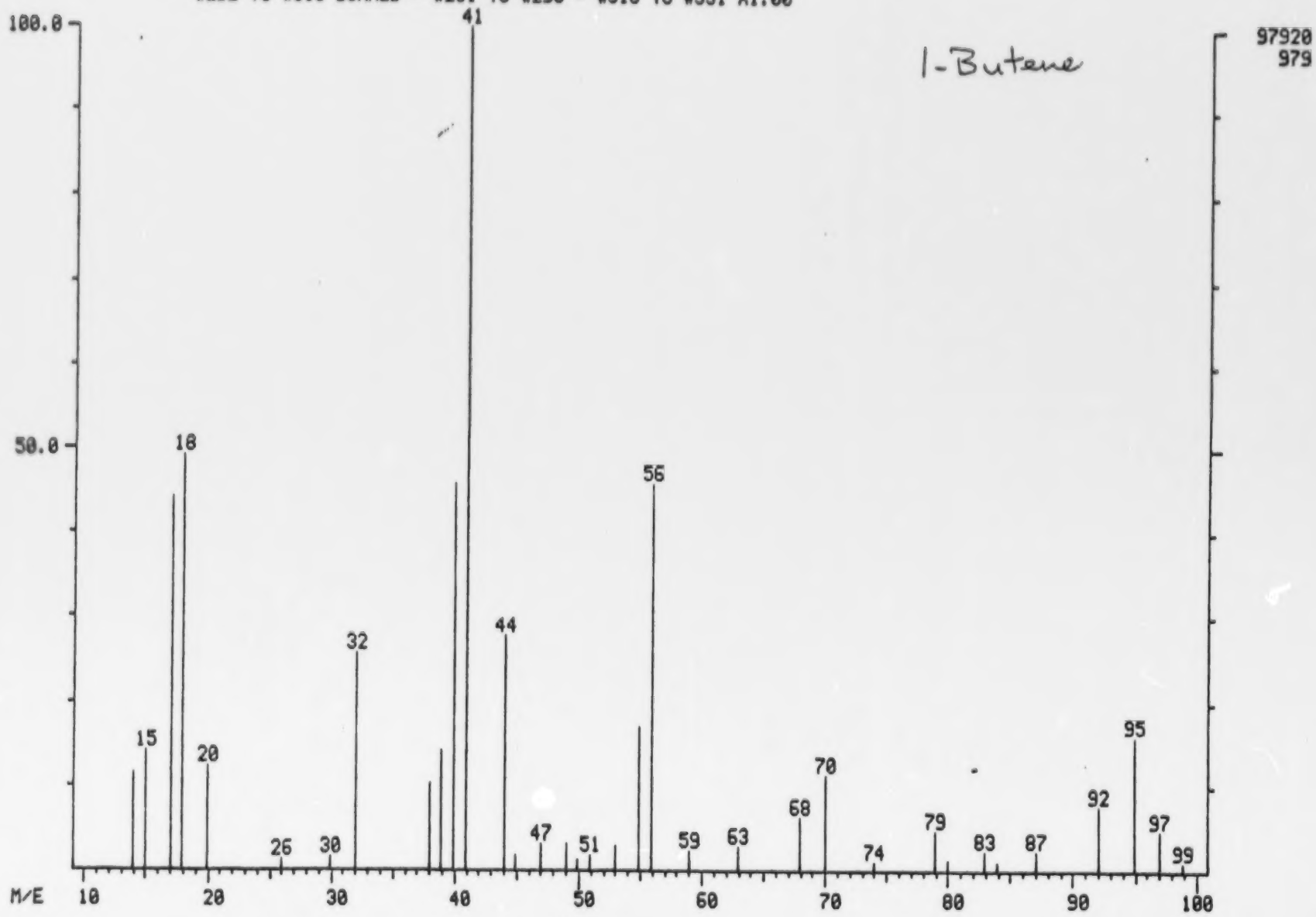
DATA: WHH27 #167
CALI: C410 #2

BASE M/E: 18
RIC: 2170870.




MASS SPECTRUM
10/04/82 15:36:00 + 6:16
SAMPLE: HC27
#180 TO #196 SUMMED - #169 TO #178 - #198 TO #211 X1.00
DATA: WHH27 #188
CALI: C410 #2
BASE M/E: 28
RIC: 675840.
Butane
100.0
50.0
202496
1012
M/E
20
40
60
80
100
120
140
28
43
58
15
51
64
92
97
12
36
47
68
76
81
108
119
128
141


MASS SPECTRUM
10/04/82 15:36:00 + 8:46
SAMPLE: HC27
#257 TO #270 SUMMED - #224 TO #243 - #285 TO #298 X1.00
DATA: WHH27 #263
CALI: C410 #2
BASE M/E: 41
RIC: 163072.
2- Butene
= 2-Methyl-1-Propene
100.0
50.0
29216
292
M/E
41
56
55
76
93
84
71
74
80
63
37
42
40
53
59
62
66
68
78
88
90
91
97
40
50
60
70
80
90
100

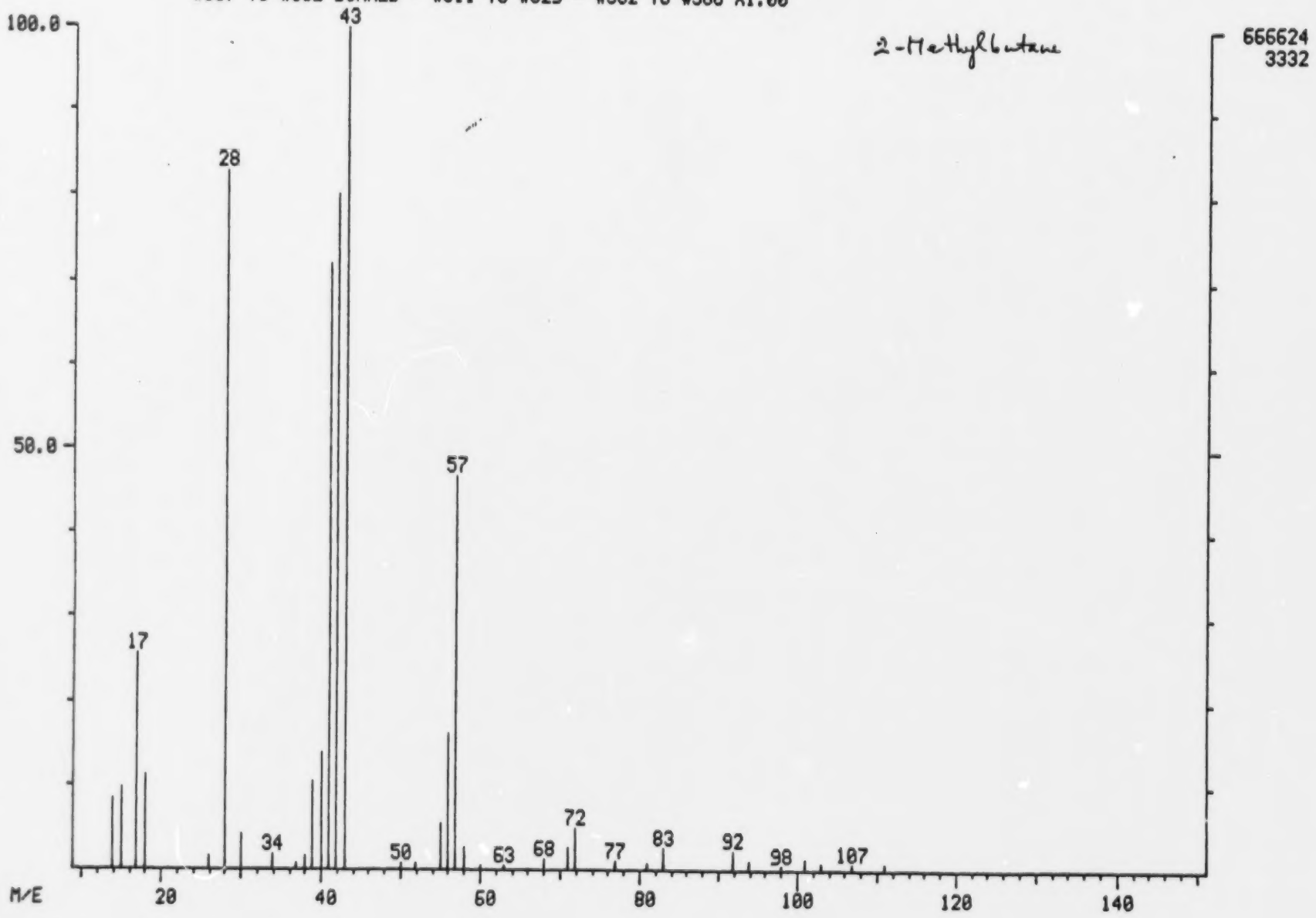

MASS SPECTRUM
10/04/82 15:36:00 + 10:08
SAMPLE: HC27
#292 TO #316 SUMMED - #281 TO #298 - #318 TO #331 X1.00
DATA: WHH27 #304
CAL1: C410 #2
BASE M/E: 41
RIC: 518656.
1-Butene
100.0
50.0
97920
979
M/E
10
20
30
40
50
60
70
80
90
100
41
18
56
44
32
15
20
26
30
47
51
59
63
68
70
74
79
83
87
92
95
97
99

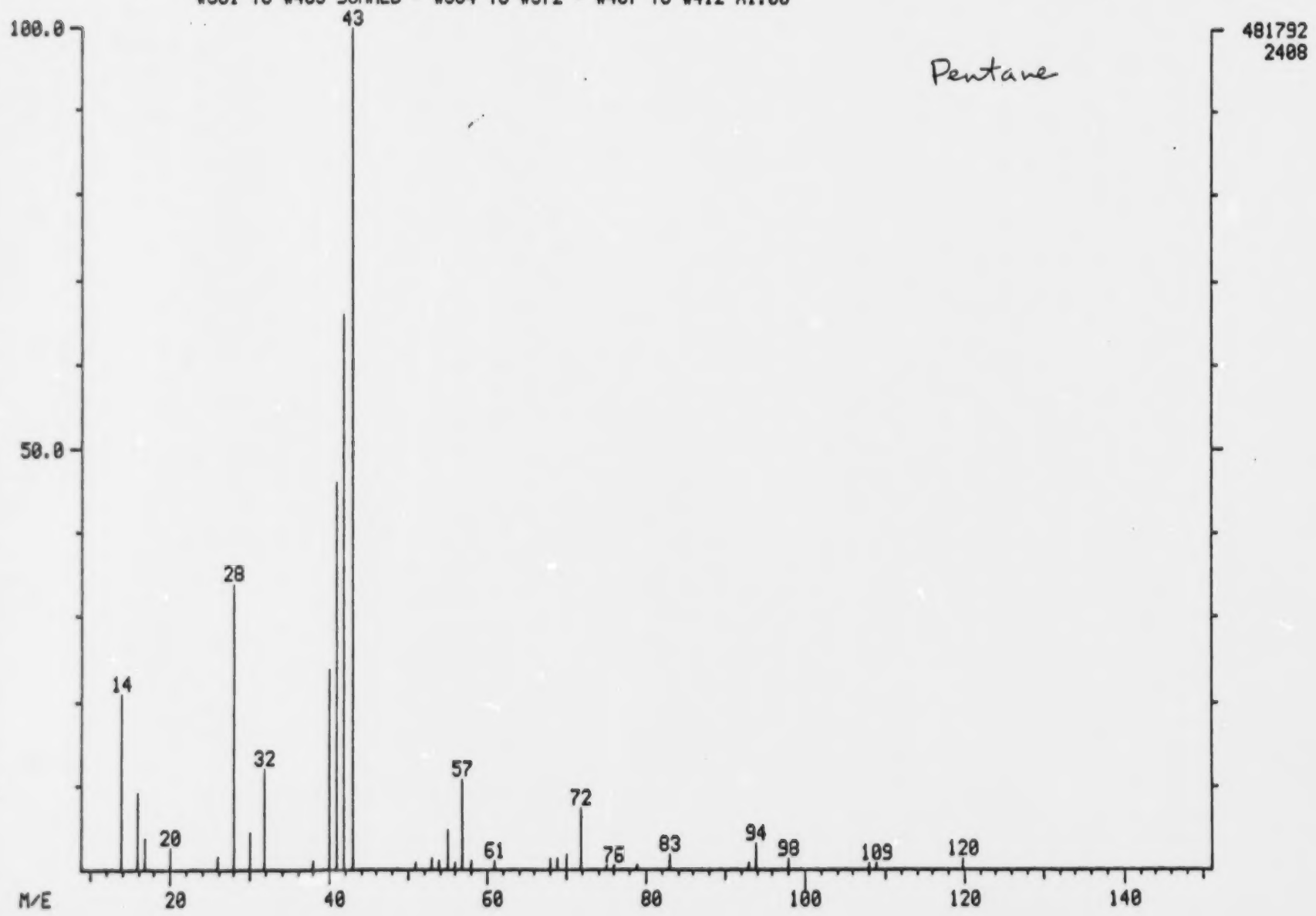
MASS SPECTRUM
10/04/82 15:36:00 + 11:38
SAMPLE: HC27
#337 TO #362 SUMMED - #311 TO #329 - #362 TO #368 X1.00

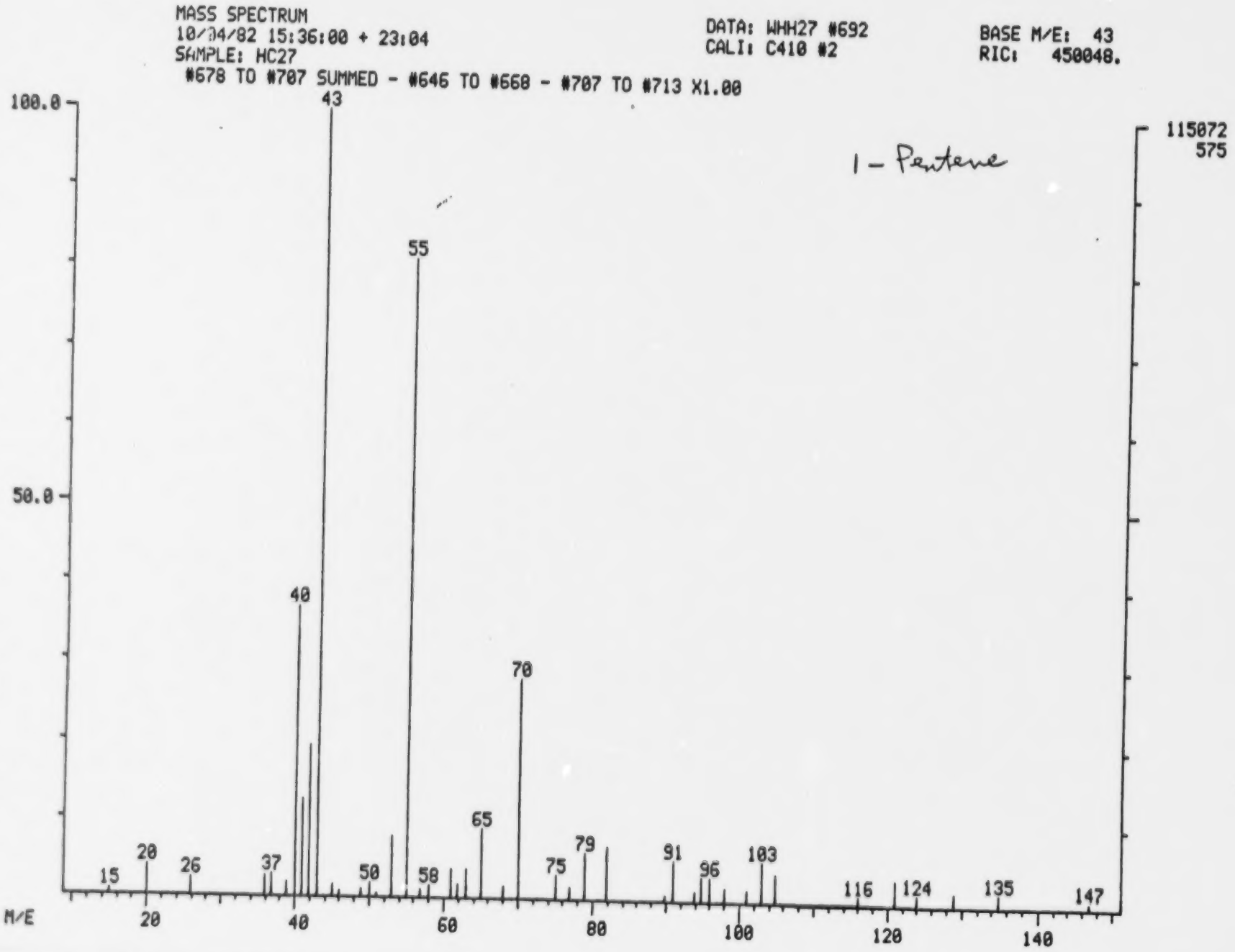
DATA: WHH27 #349
CALI: C410 #2

BASE M/E: 43
RIC: 3481590.

2-Methylbutane



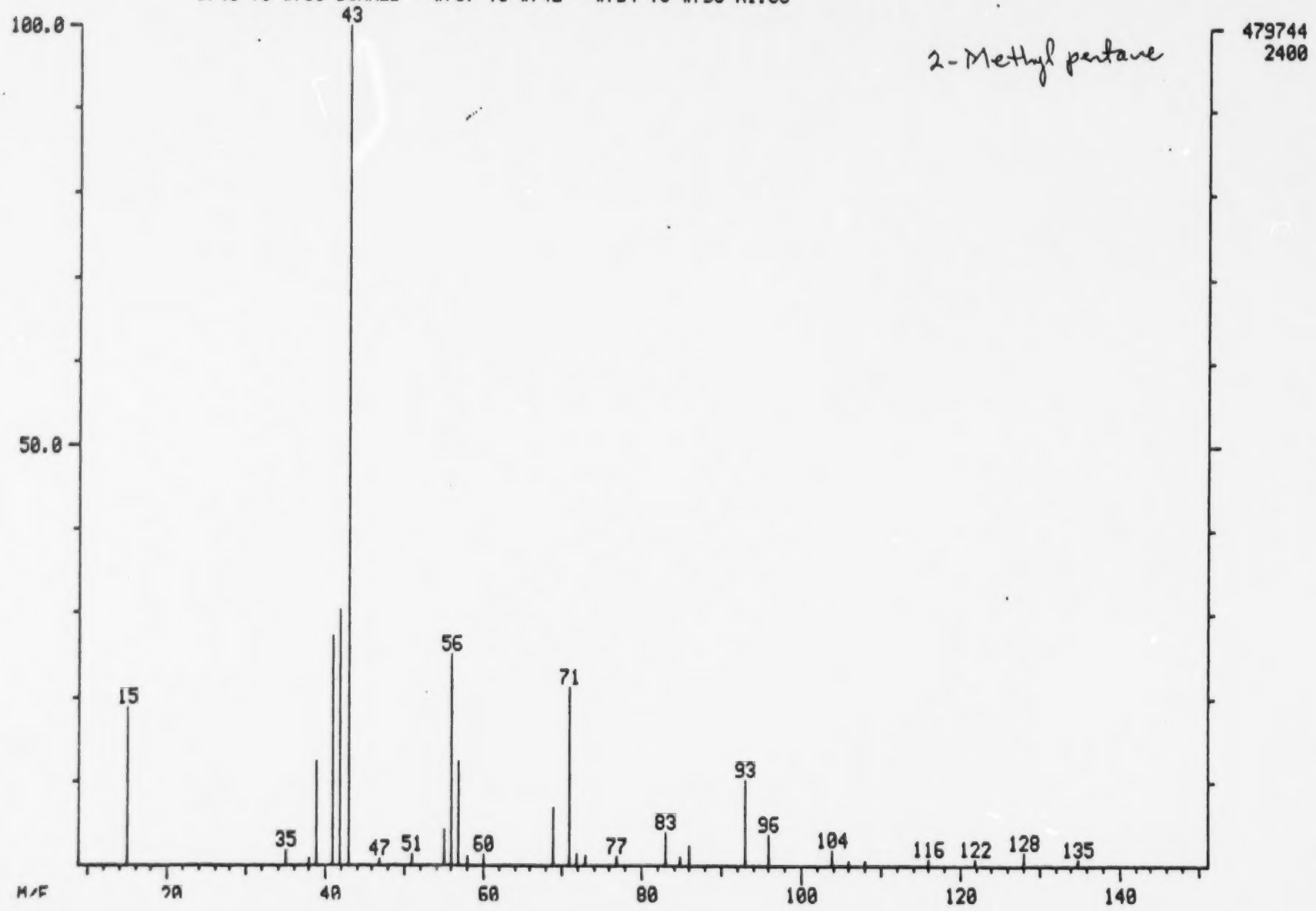

MASS SPECTRUM
10/04/82 15:36:00 + 13:04
SAMPLE: HC27
#381 TO #403 SUMMED - #364 TO #372 - #407 TO #412 X1.00
DATA: WHH27 #392
CALI: C410 #2
BASE M/E: 43
RIC: 1796090.
Pentane
100.0
50.0
481792
2408
43
28
14
32
57
72
20
61
75
83
94
98
109
120
M/E
20
40
60
80
100
120
140


MASS SPECTRUM
10/04/82 15:36:00 + 23:04
SAMPLE: HC27
#678 TO #707 SUMMED - #646 TO #668 - #707 TO #713 X1.00
DATA: WHH27 #692
CALI: C410 #2
BASE M/E: 43
RIC: 450048.
1 - Pentene
115072
575
100.0
50.0
43
55
40
70
65
79
91
103
20
26
37
50
58
75
96
116
124
135
147
15
M/E
20
40
60
80
100
120
140

MASS SPECTRUM
10/04/82 15:36:00 + 25:32
SAMPLE: HC27
#748 TO #785 SUMMED - #737 TO #742 - #794 TO #798 X1.00

DATA: WHH27 #766
CALI: C410 #2

BASE M/E: 43
RIC: 1456120.

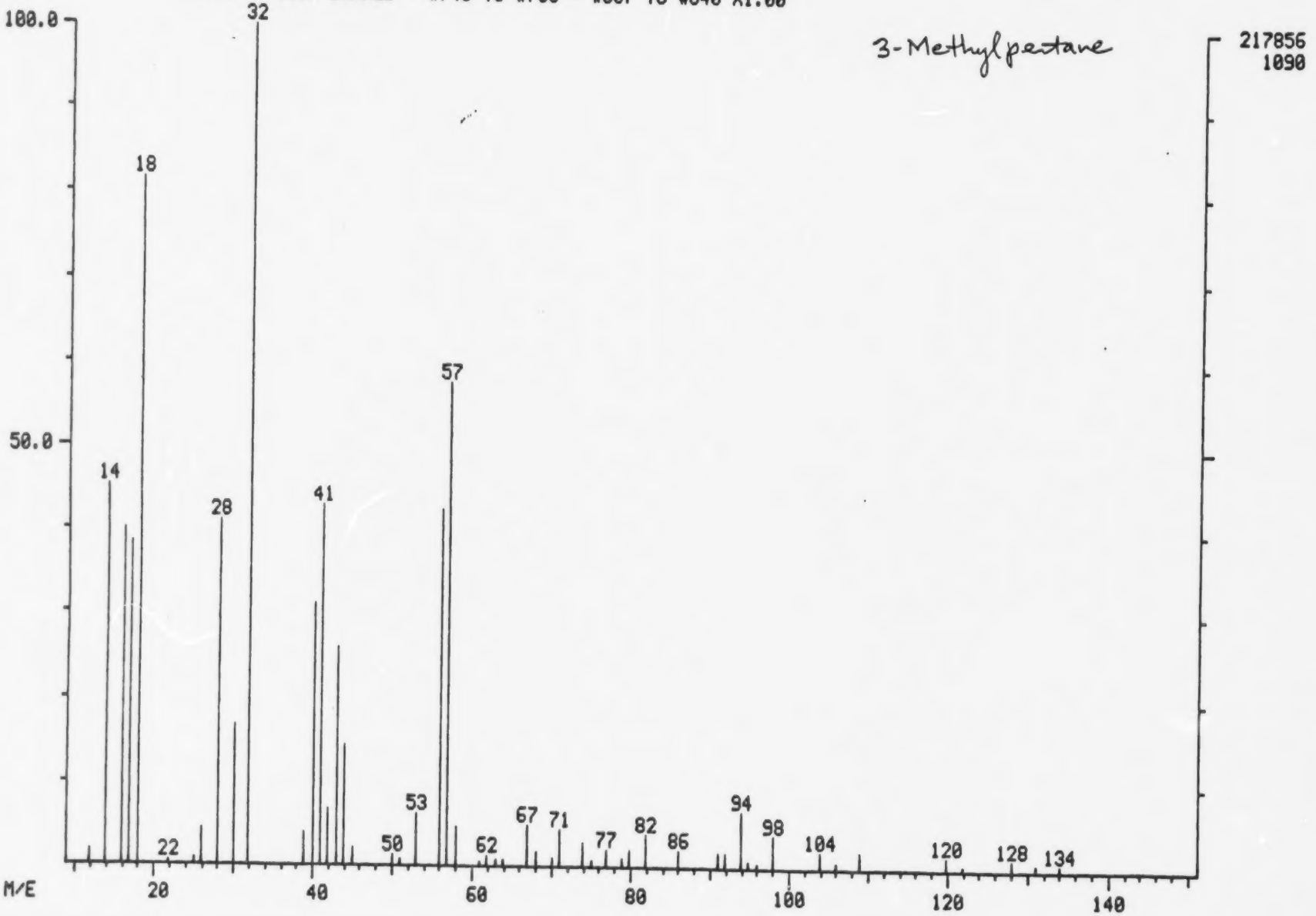


2-Methyl pentane

MASS SPECTRUM
10/04/82 15:36:00 + 26:20
SAMPLE: HC27
#774 TO #807 SUMMED - #748 TO #766 - #807 TO #840 X1.00

DATA: WHH27 #790
CALI: C410 #2

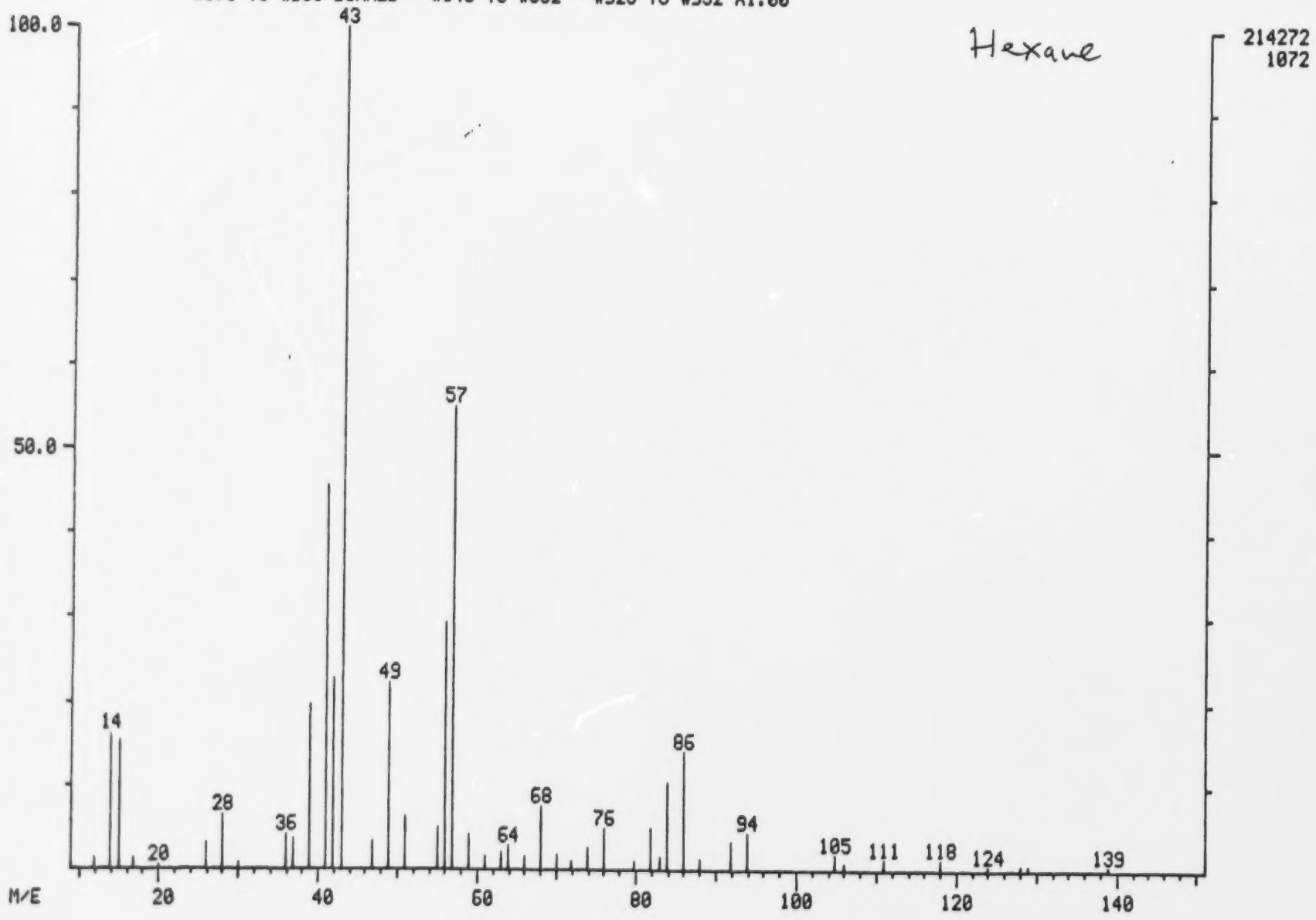
BASE M/E: 32
RIC: 1462270.

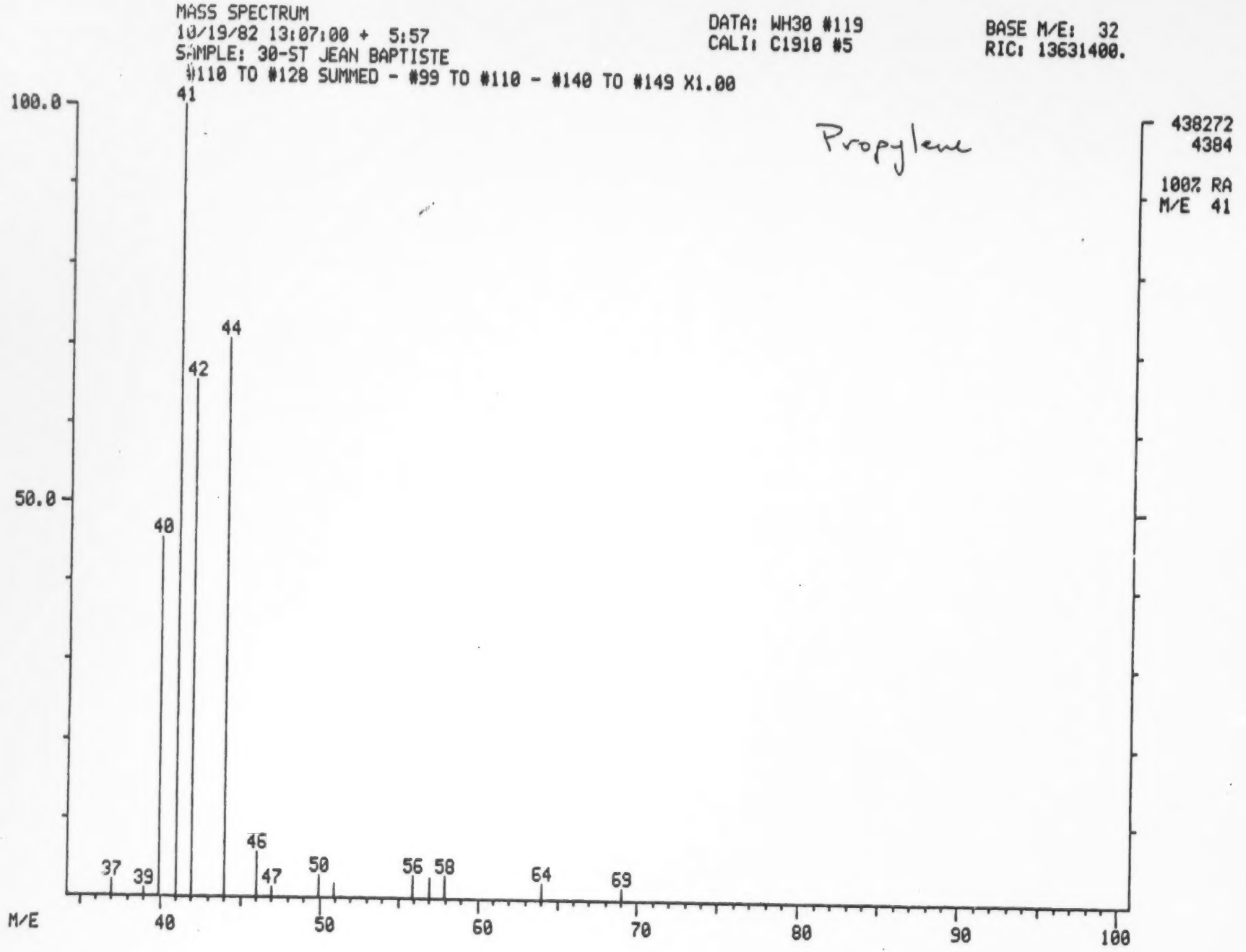


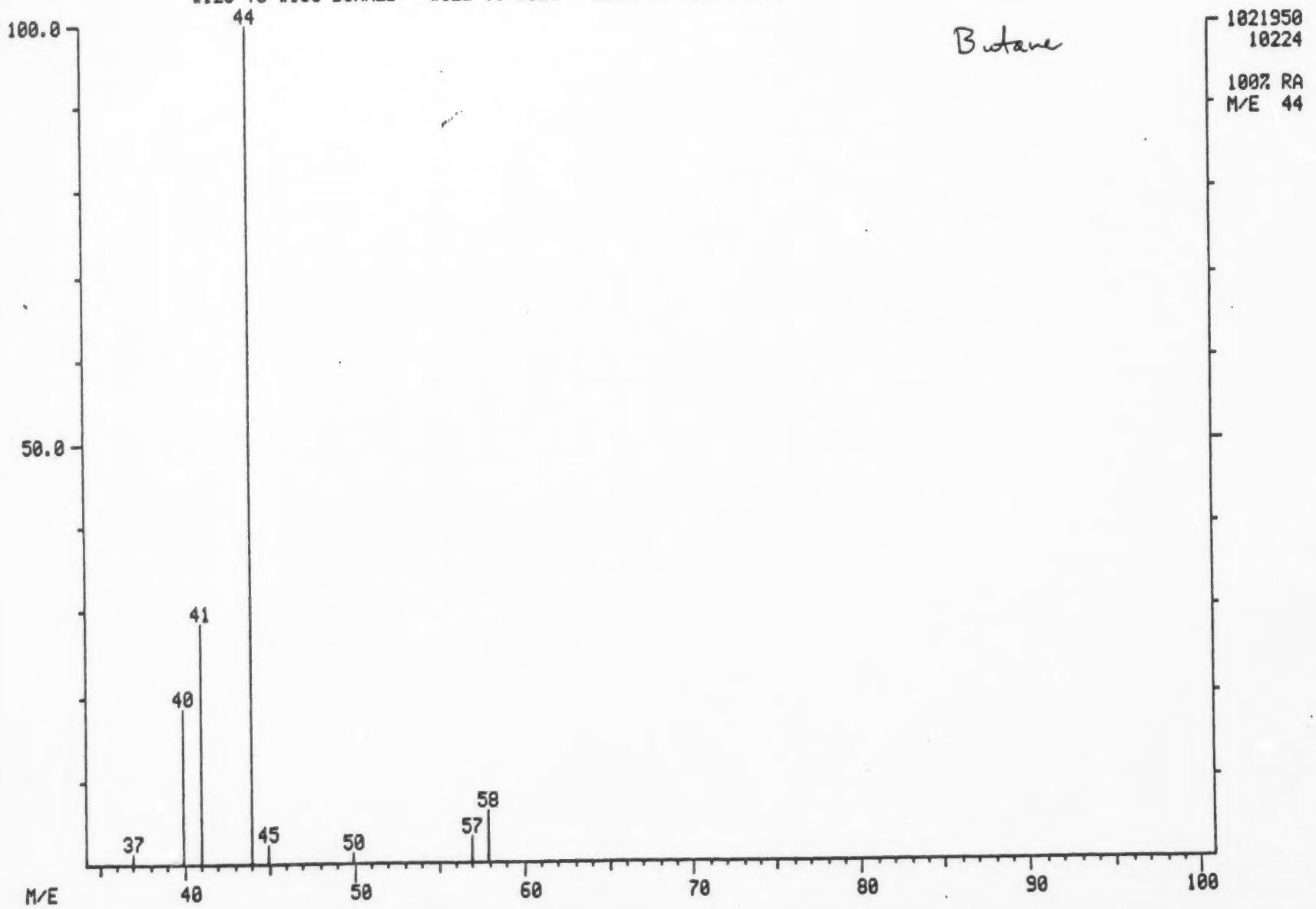
MASS SPECTRUM
10/04/82 15:36:00 + 29:42
SAMPLE: HC27
#875 TO #908 SUMMED - #840 TO #862 - #925 TO #932 X1.00

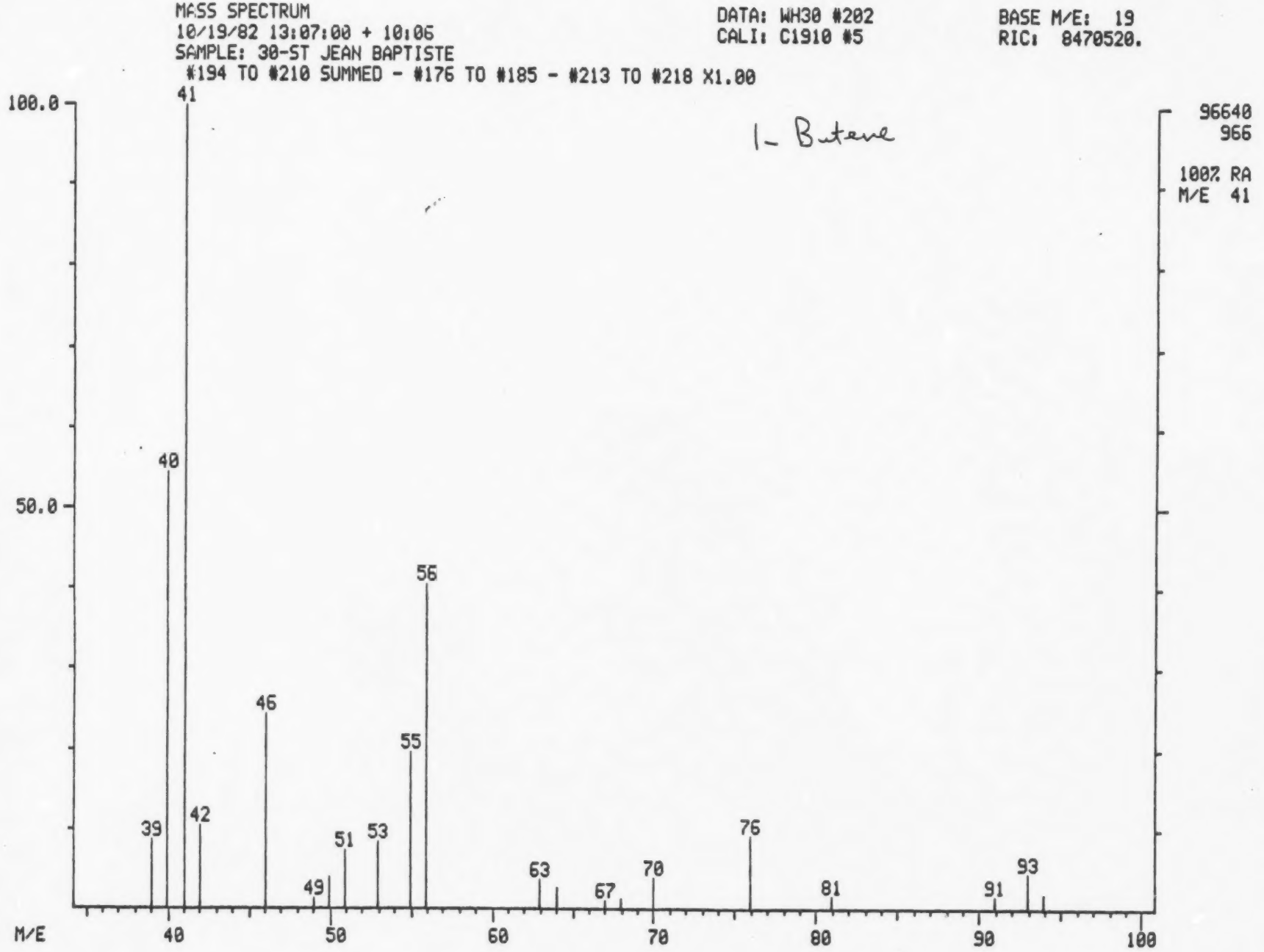
DATA: WHH27 #891
CALI: C410 #2

BASE M/E: 43
RIC: 962560.




MASS SPECTRUM
10/19/82 13:07:00 + 5:57
SAMPLE: 30-ST JEAN BAPTISTE
#110 TO #128 SUMMED - #99 TO #110 - #140 TO #149 X1.00
DATA: WH30 #119
CALI: C1910 #5
BASE M/E: 32
RIC: 13631400.
Propylene
438272
4384
100% RA
M/E 41
100.0
50.0
41
44
42
40
46
37
39
47
50
56
58
64
69
M/E
40
50
60
70
80
90
100


MASS SPECTRUM
10/19/82 13:07:00 + 6:30
SAMPLE: 30-ST JEAN BAPTISTE
#126 TO #135 SUMMED - #122 TO #125 - #140 TO #143 X1.00
DATA: WH30 #130
CALI: C1910 #5
BASE M/E: 33
RIC: 7864310.
Butane
1021950
10224
100% RA
M/E 44
100.0
50.0
M/E
44
41
40
37
45
50
57
58
40
50
60
70
80
90
100

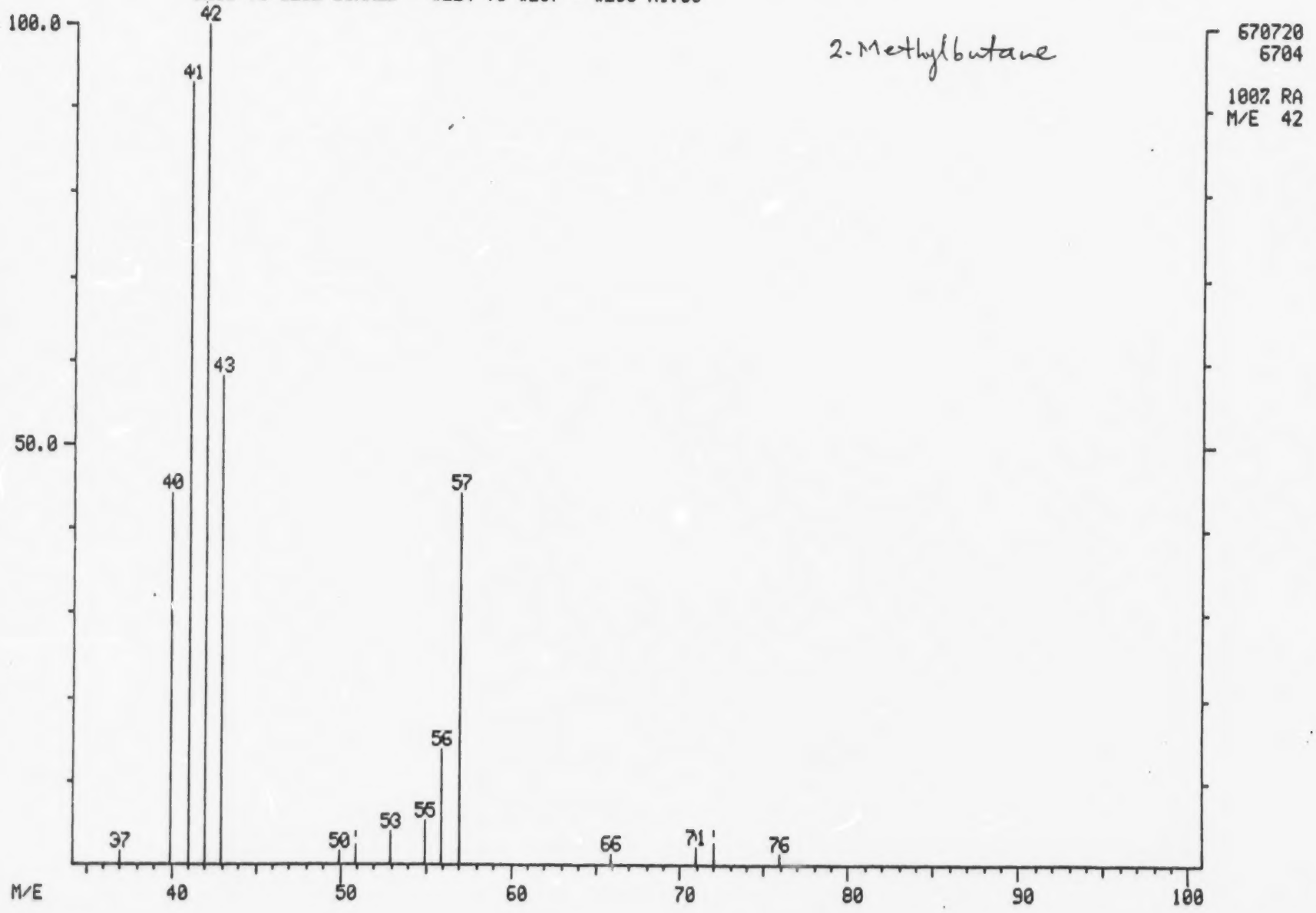

MASS SPECTRUM
10/19/82 13:07:00 + 10:06
SAMPLE: 30-ST JEAN BAPTISTE
#194 TO #210 SUMMED - #176 TO #185 - #213 TO #218 X1.00
DATA: WH30 #202
CALI: C1910 #5
BASE M/E: 19
RIC: 8470520.
1- Butene
96640
966
100% RA
M/E 41
100.0
50.0
M/E
40
50
60
70
80
90
100
41
40
39
42
46
49
51
53
55
56
63
67
70
76
81
91
93

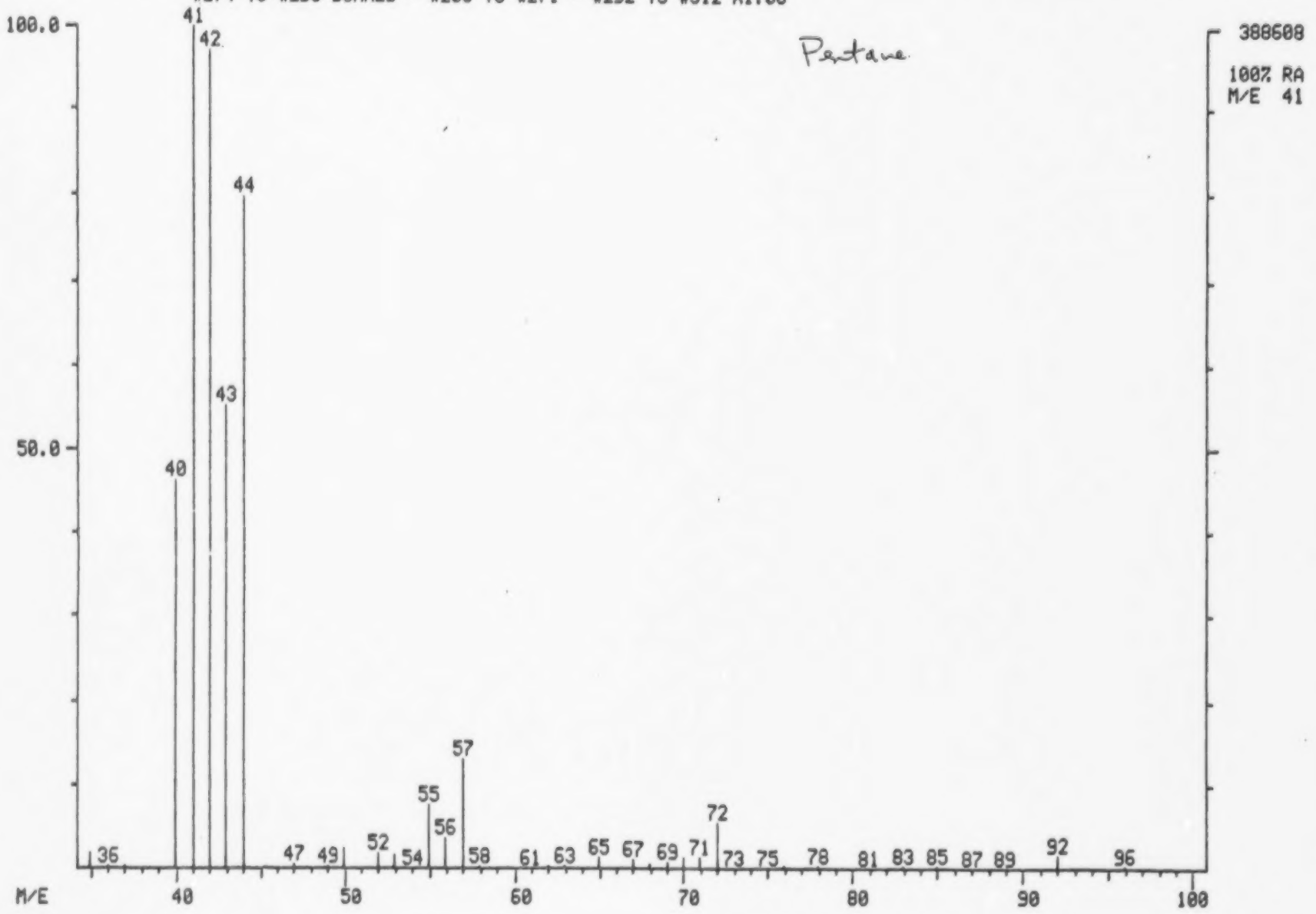
MASS SPECTRUM
10/19/87 13:07:00 + 12:30
SAMPLE: 30-ST JEAN BAPTISTE
#238 TO #262 SUMMED - #224 TO #237 - #235 X1.00

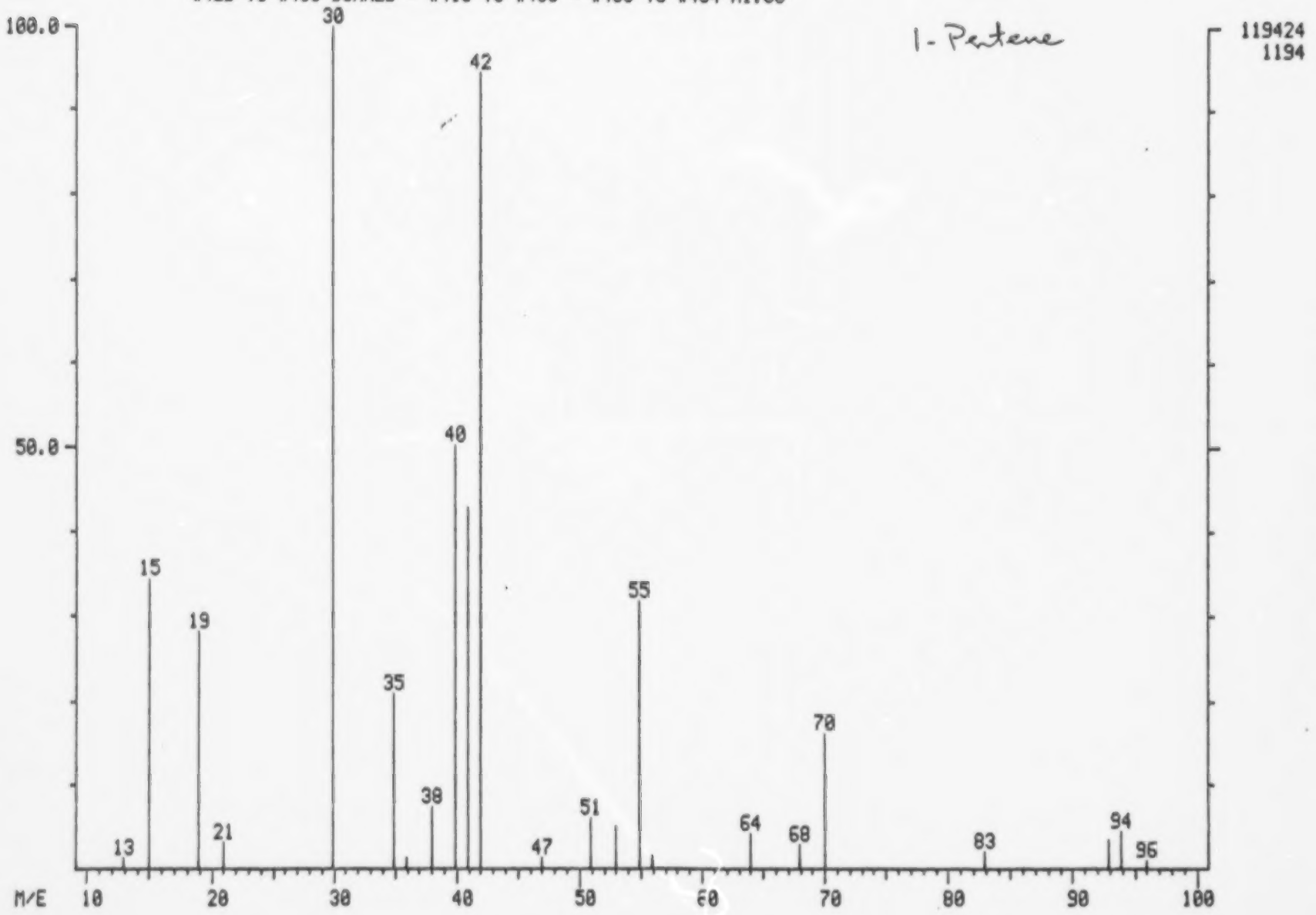
DATA: WH30 #250
CALI: C1910 #5

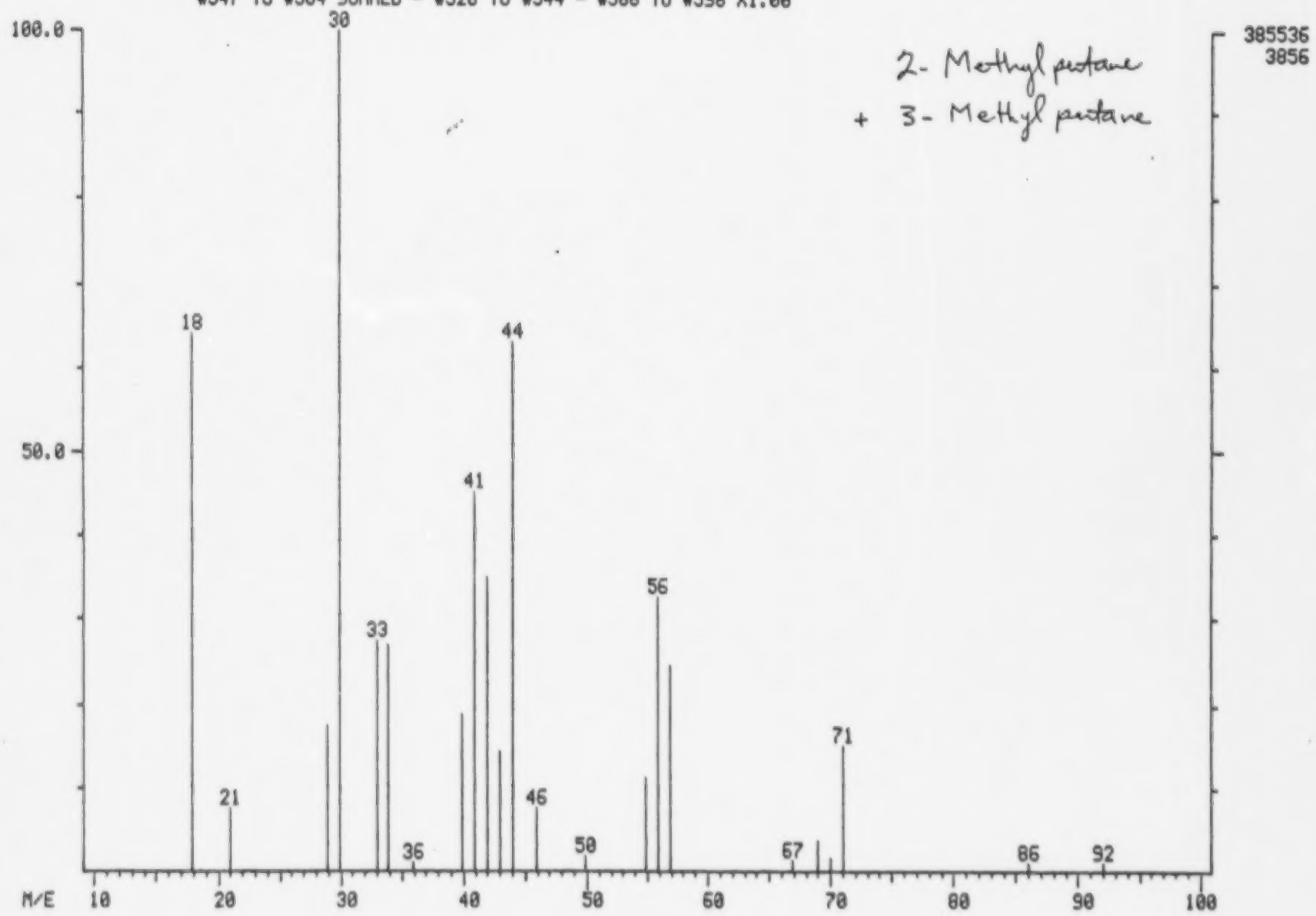
BASE M/E: 33
RIC: 8601590.

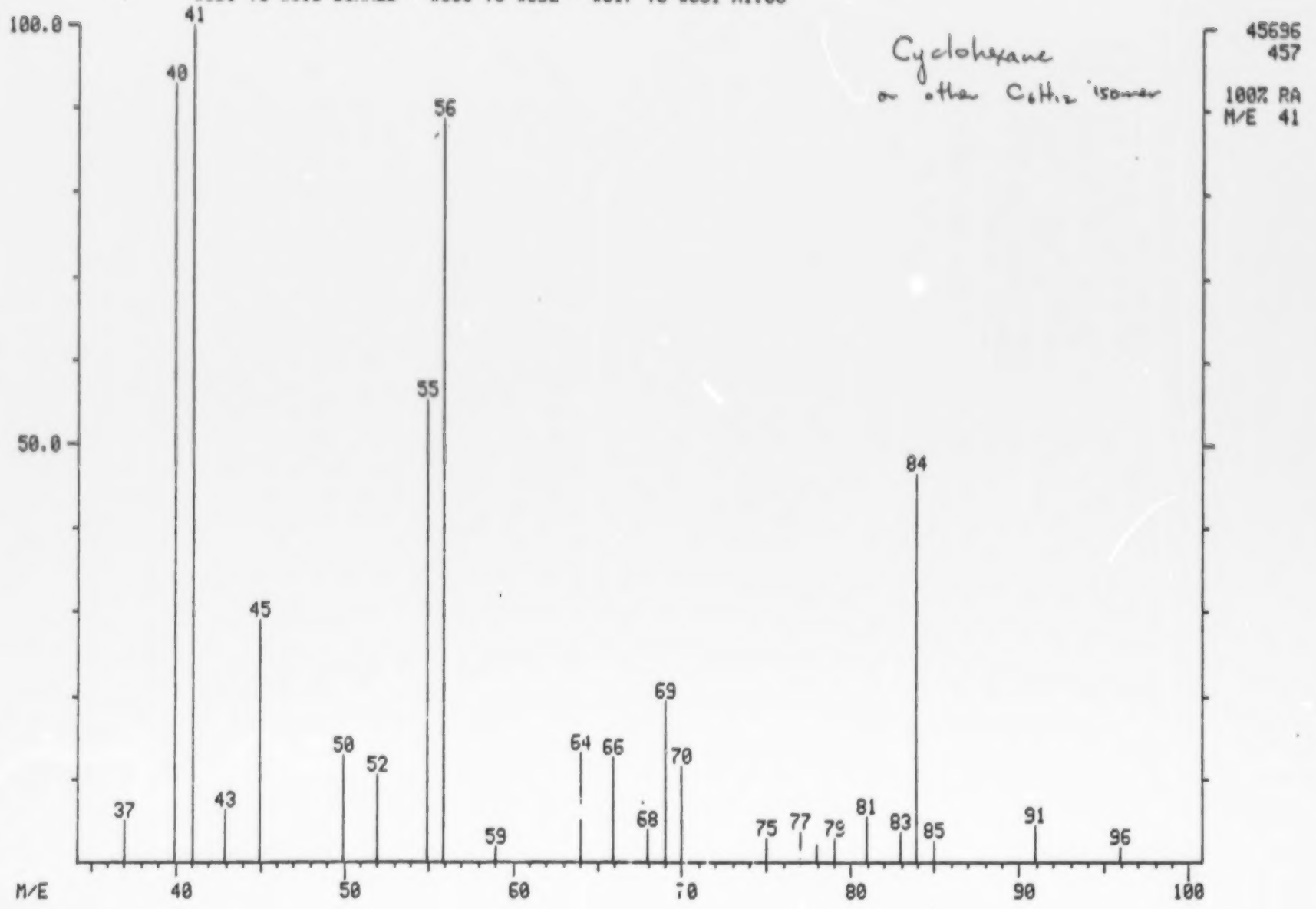
2-Methylbutane




MASS SPECTRUM
10/19/82 13:07:00 + 14:06
SAMPLE: 30-ST JEAN BAPTISTE
#274 TO #290 SUMMED - #260 TO #271 - #292 TO #312 X1.00
DATA: WH30 #282
CALI: C1910 #5
BASE M/E: 32
RIC: 3895290.
Pentane
388608
100% RA
M/E 41
100.0
50.0
M/E
41
42
44
43
40
57
55
56
72
35
47
49
52
54
58
61
63
65
67
69
71
73
75
78
81
83
85
87
89
92
96
40
50
60
70
80
90
100


MASS SPECTRUM
10/19/82 13:07:00 + 22:03
SAMPLE: 30-ST JEAN BAPTISTE
#429 TO #453 SUMMED - #413 TO #435 - #453 TO #464 X1.00
DATA: WH30 #441
CALI: C1910 #5
BASE M/E: 30
RIC: 563200.
1-Pentene
119424
1194
100.0
50.0
M/E
10
20
30
40
50
60
70
80
90
100
13
15
19
21
30
35
38
40
42
47
51
55
64
68
70
83
94
96

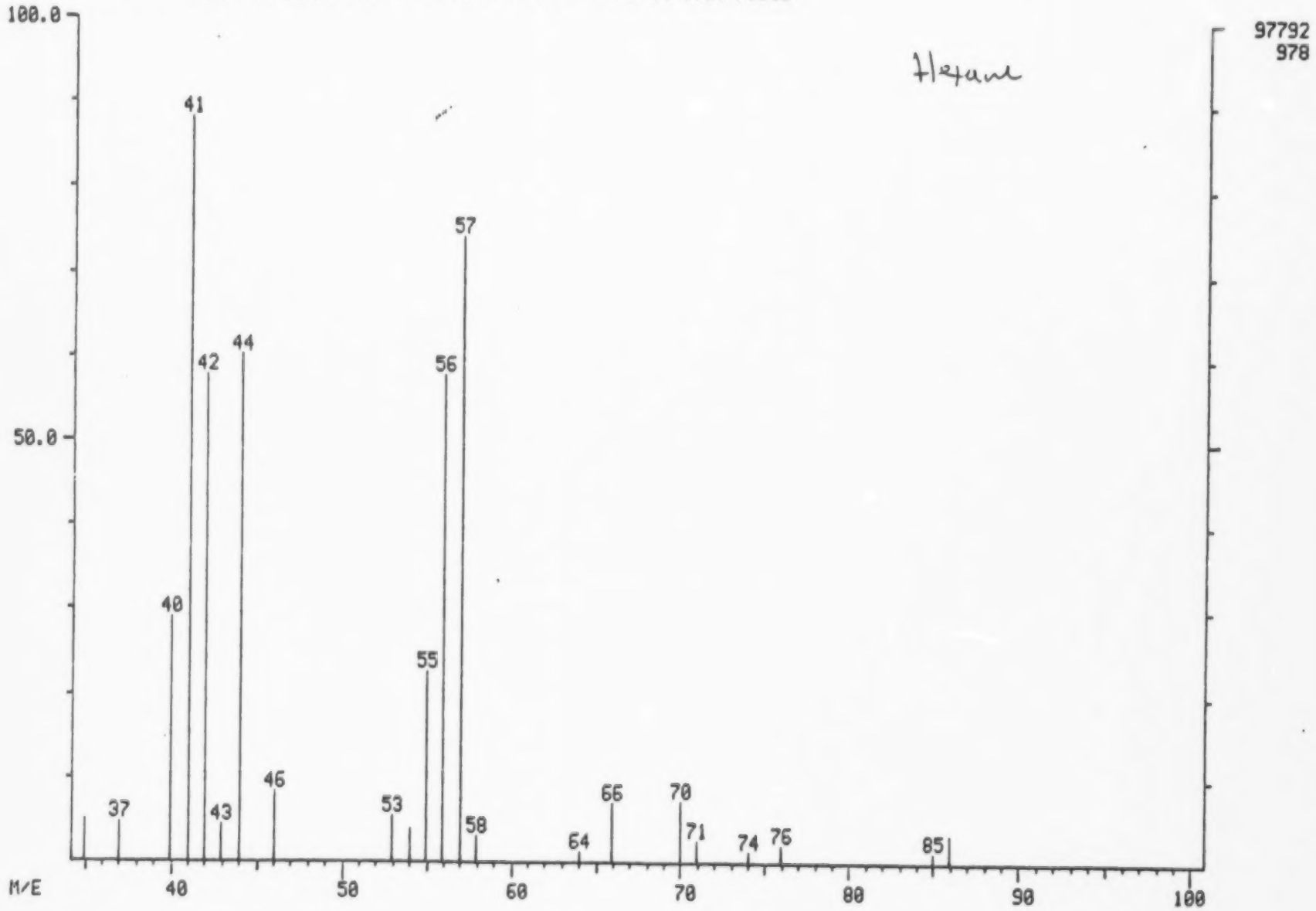

MASS SPECTRUM
10/19/82 13:07:00 + 28:15
SAMPLE: 30-ST JEAN BAPTISTE
#547 TO #584 SUMMED - #528 TO #544 - #586 TO #598 X1.00
DATA: WH30 #565
CALI: C1910 #5
BASE M/E: 30
RIC: 2043900.
100.0
50.0
385536
3856
2- Methyl pentane
+ 3- Methyl pentane
18
30
44
41
56
33
71
21
46
36
50
67
86
92
M/E
10
20
30
40
50
60
70
80
90
100

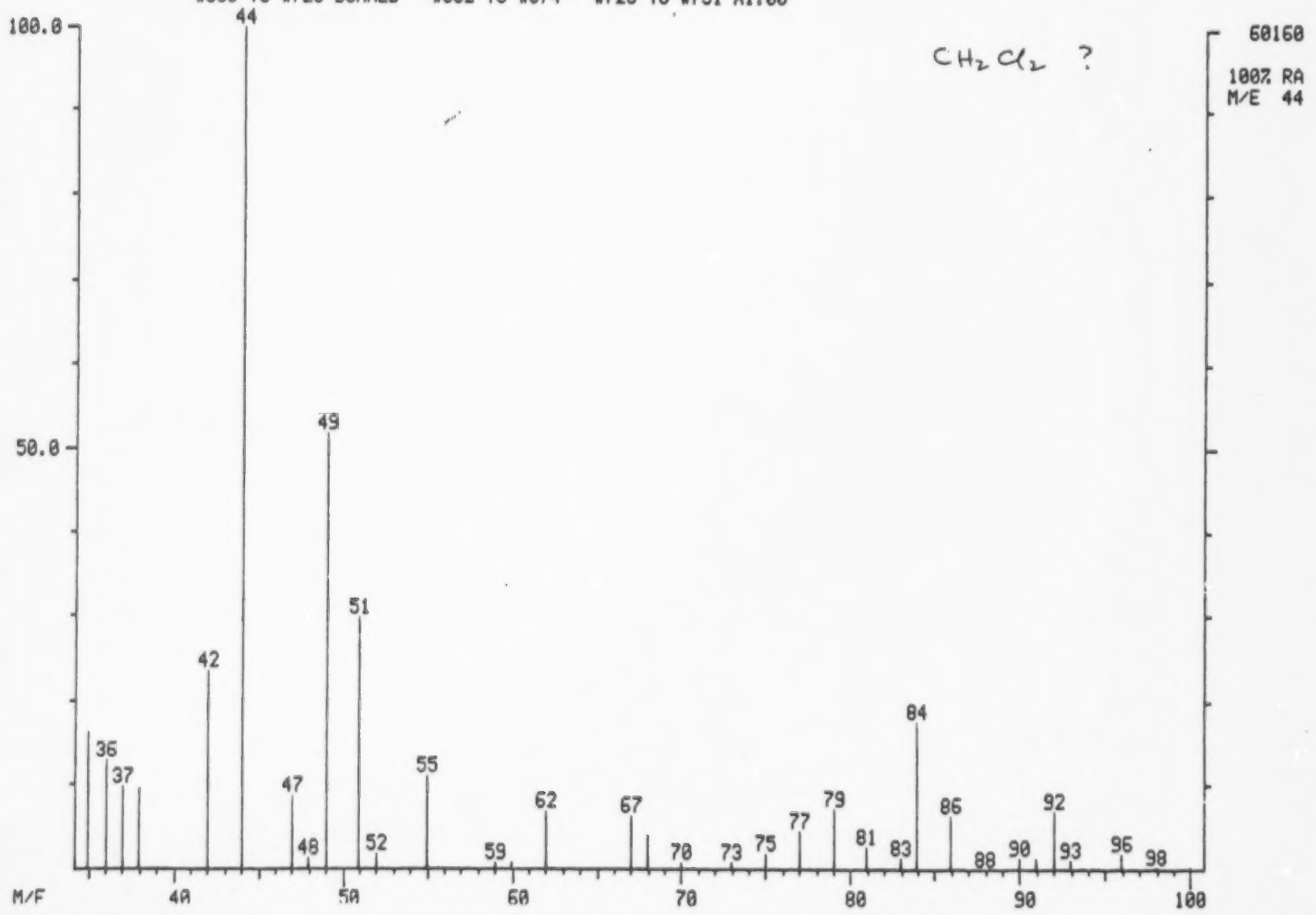

MASS SPECTRUM
10/19/82 13:07:00 + 30:15
SAMPLE: 30-ST JEAN BAPTISTE
#591 TO #619 SUMMED - #583 TO #592 - #617 TO #631 X1.00
DATA: WH30 #685
CALI: C1910 #5
BASE M/E: 18
RIC: 594944.
Cyclohexane
or other C6H12 isomer
45696
457
100% RA
M/E 41
100.0
50.0
M/E
40
50
60
70
80
90
100
41
40
56
55
84
45
69
50
52
64
66
70
37
43
68
59
75
77
79
81
83
85
91
96

MASS SPECTRUM
10/19/82 13:07:00 + 32:48
SAMPLE: 30-ST JEAN BAPTISTE
#643 TO #669 SUMMED - #619 TO #636 - #679 TO #705 X1.00

DATA: WH30 #656
CALI: C1910 #5

BASE M/E: 31
RIC: 617472.




MASS SPECTRUM
10/19/82 13:07:00 + 34:45
SAMPLE: 30-ST JEAN BAPTISTE
#668 TO #723 SUMMED - #662 TO #674 - #725 TO #751 X1.00
DATA: WH30 #695
CALI: C1910 #5
BASE M/E: 33
RIC: 2514940.
60160
100% RA
M/E 44
CH2Cl2 ?
100.0
50.0
M/E
40
50
60
70
80
90
100
44
49
51
42
36
37
47
48
52
55
59
62
67
70
73
75
77
79
81
83
84
86
88
90
92
93
96
98

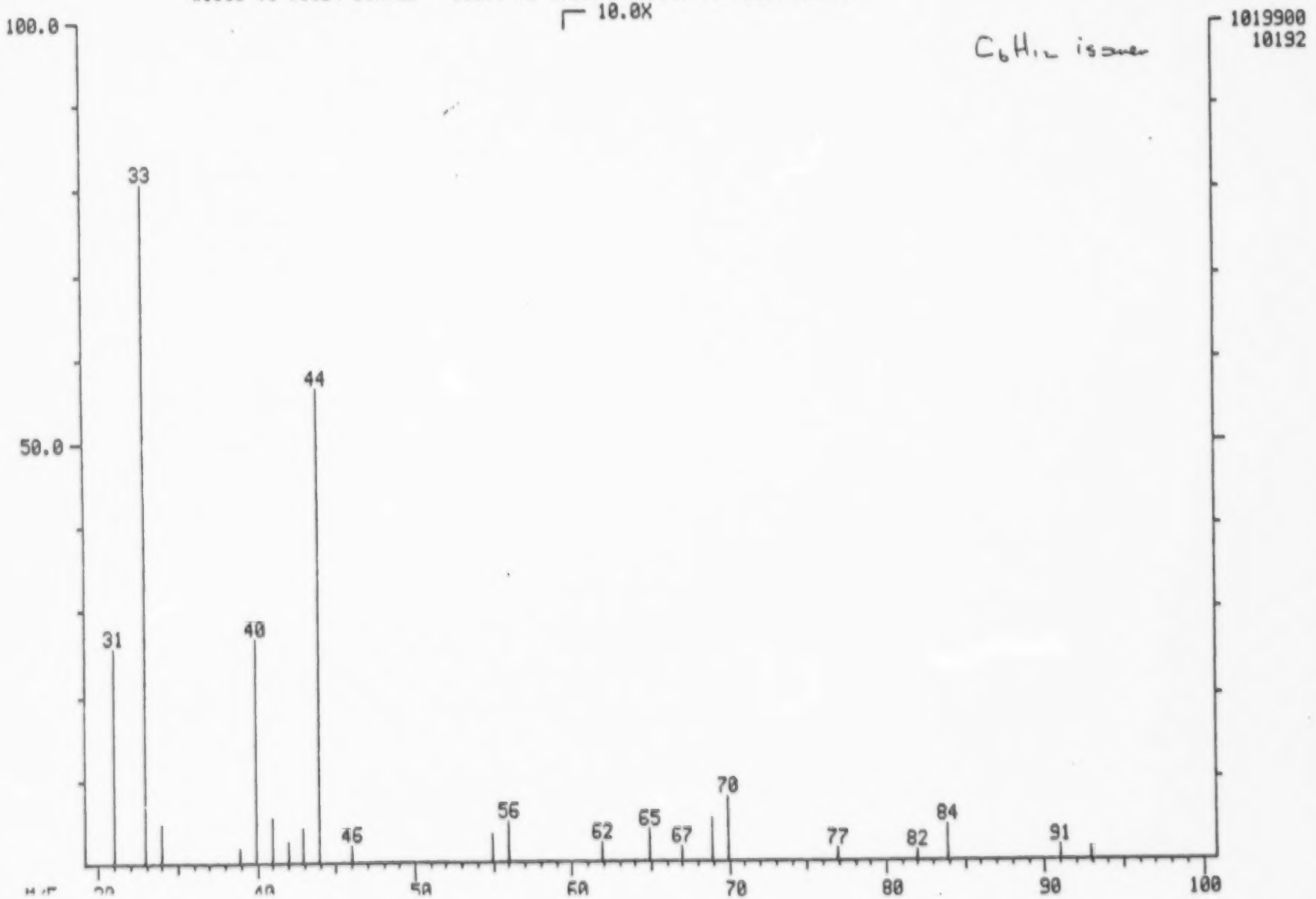
MASS SPECTRUM
10/19/82 13:07:00 + 53:48
SAMPLE: 30-ST JEAN BAPTISTE
#1058 TO #1094 SUMMED - #1031 TO #1050 - #1103 TO #1115 X1.00

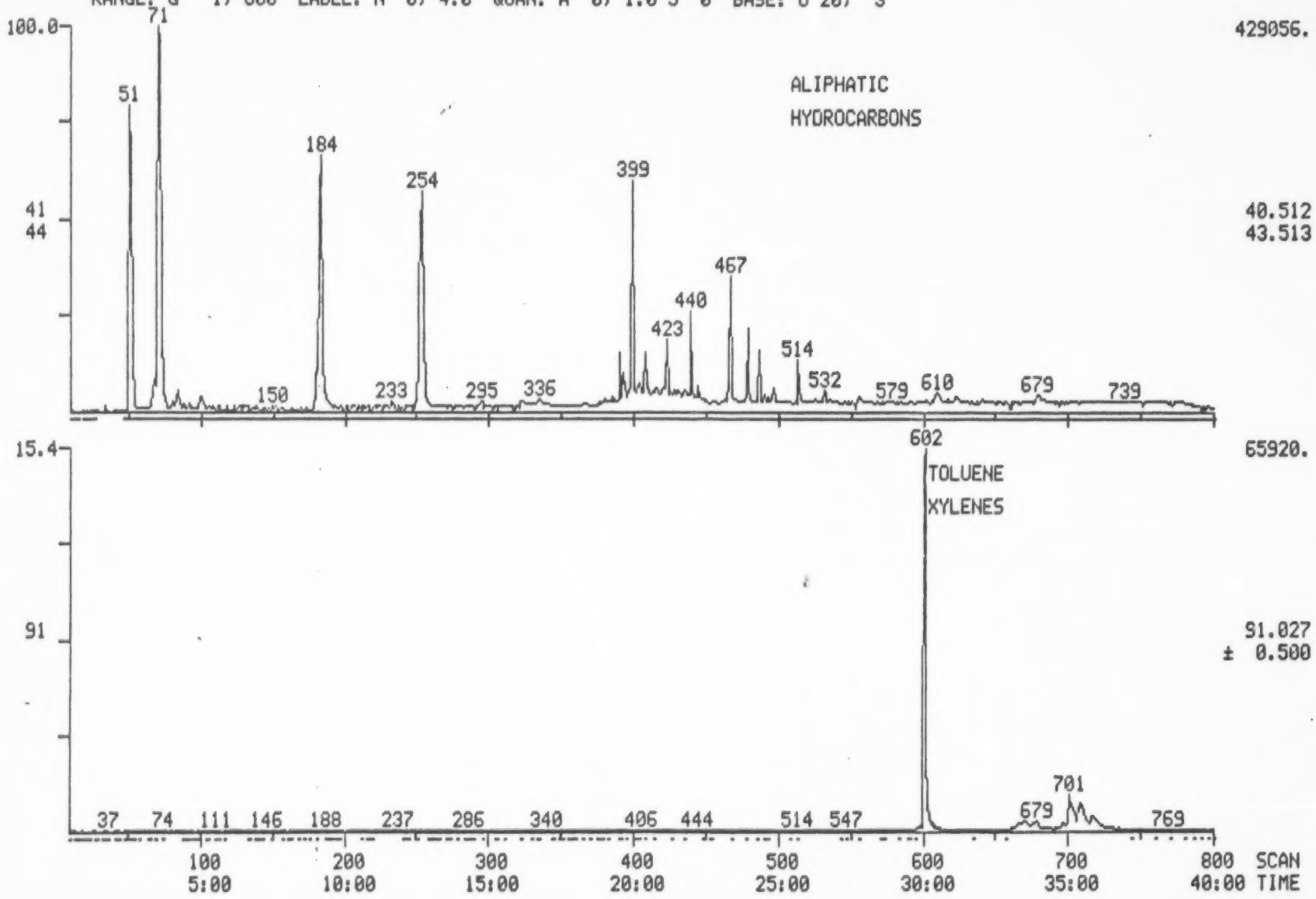
DATA: WH30 #1076
CALI: C1910 #5

BASE M/E: 29
RIC: 5160950.

10.0X

C_6H_{12} isomer



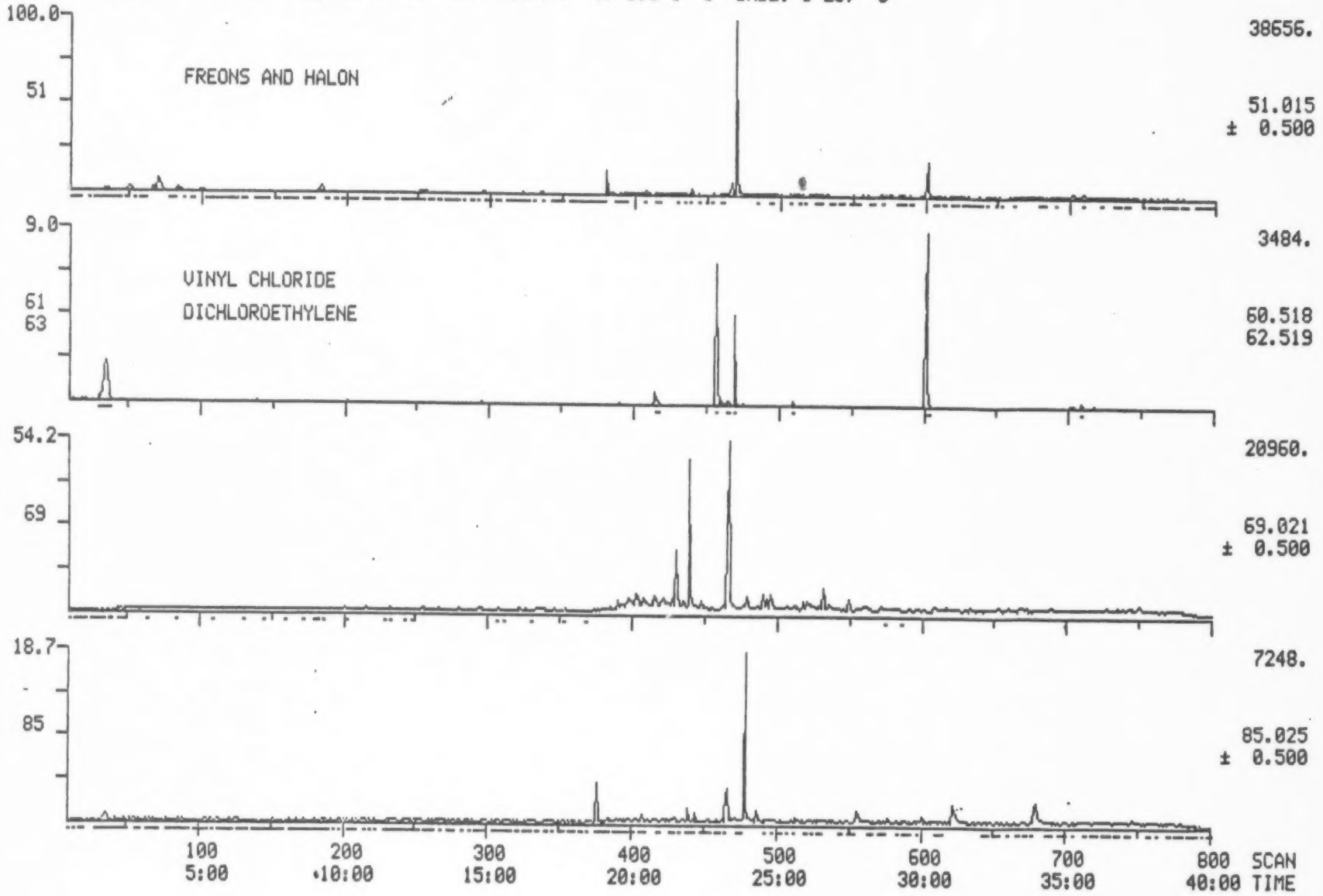

MASS CHROMATOGRAMS
12/08/82 15:10:00
SAMPLE: ST JEAN BAPTISTE HC-62
CONDS.: 30M SE54,-20 DEG
RANGE: G 1, 800 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
DATA: WH621 #1
CALI: C8121 #1
SCANS 10 TO 800
FIGURE I
100.0
429056.
ALIPHATIC
HYDROCARBONS
71
51
184
254
399
467
440
423
514
532
579
610
679
739
150
233
295
336
41
44
40.512
43.513
15.4
65920.
602
TOLUENE
XYLENES
91
91.027
± 0.500
701
679
769
37 74 111 146 188 237 286 340 406 444 514 547
100
5:00
200
10:00
300
15:00
400
20:00
500
25:00
600
30:00
700
35:00
800
40:00
SCAN
TIME

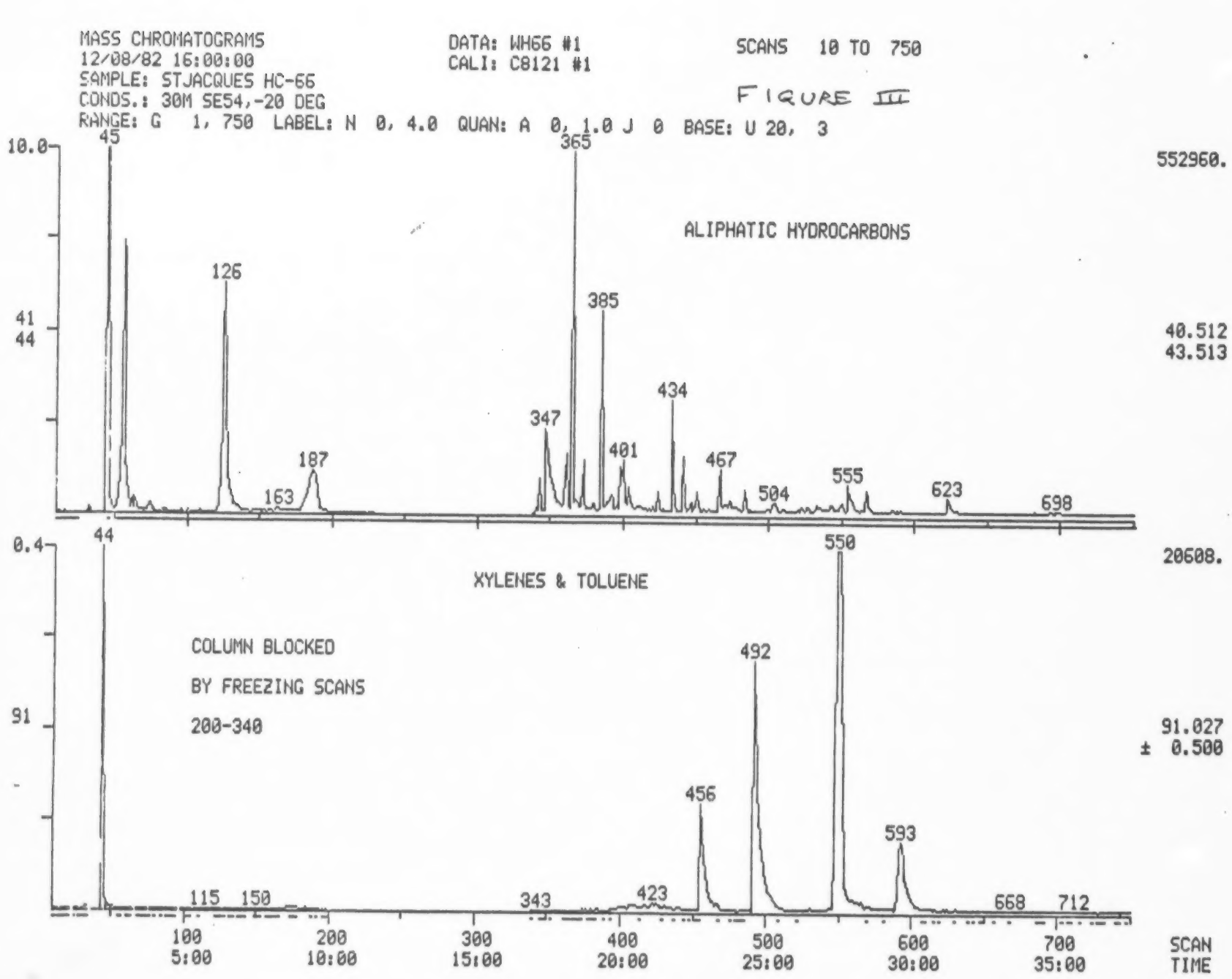
MASS CHROMATOGRAMS
12/08/82 15:10:00
SAMPLE: ST JEAN BAPTISTE HC-62
CONDS.: 30M SE54,-20 DEG
RANGE: G 1, 800 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3

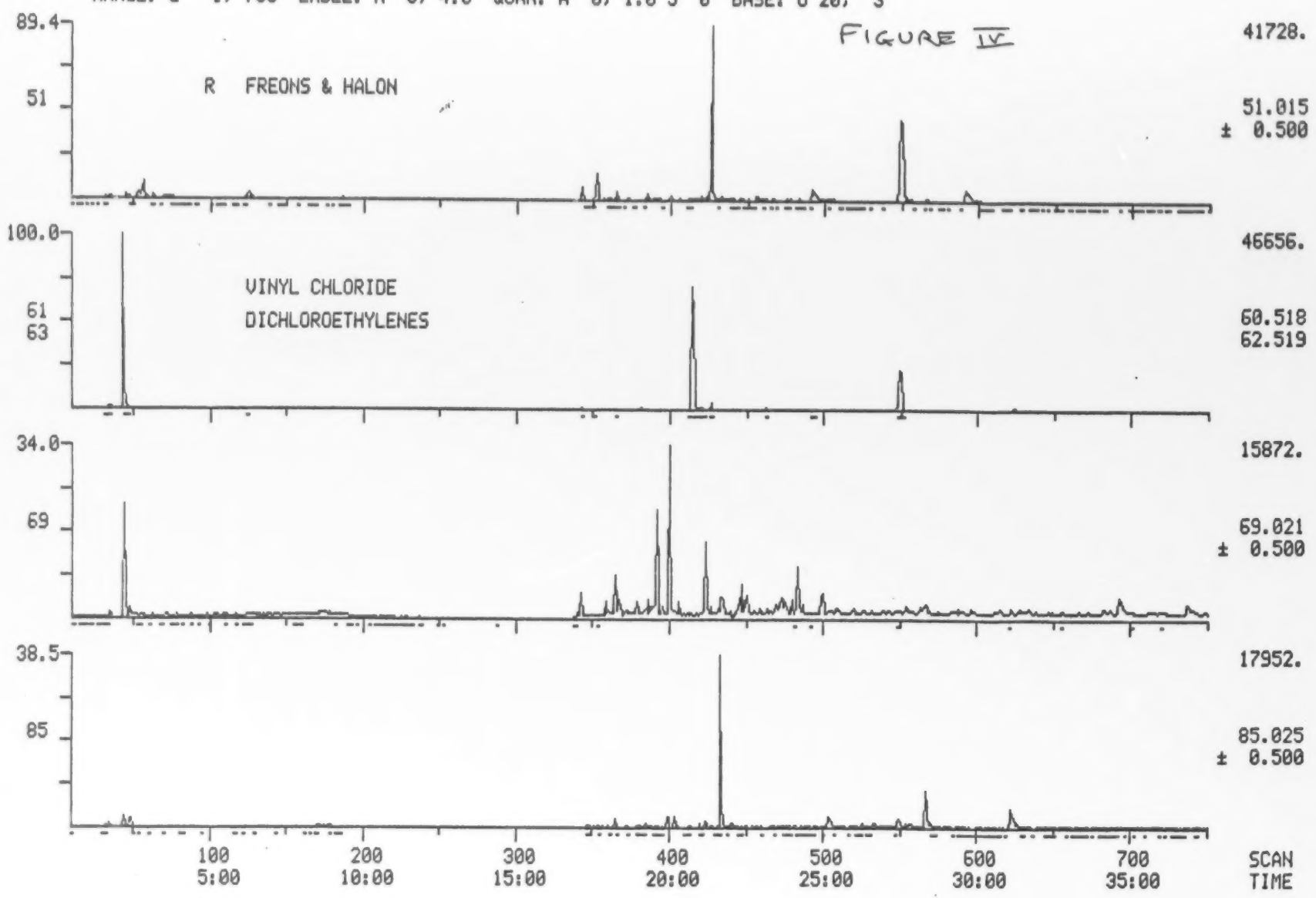
DATA: WH621 #377
CALI: C8121 #1

SCANS 10 TO 800

FIGURE II




MASS CHROMATOGRAMS
12/08/82 16:00:00
SAMPLE: STJACQUES HC-66
CONDS.: 30M SE54,-20 DEG
RANGE: G 1, 750 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
DATA: WH66 #1
CALI: C8121 #1
SCANS 10 TO 750
FIGURE III
10.0
41
44
45
126
163
187
347
365
385
401
434
467
504
555
623
698
552960.
ALIPHATIC HYDROCARBONS
40.512
43.513
0.4
91
44
115
150
343
423
456
492
550
593
668
712
20608.
XYLENES & TOLUENE
COLUMN BLOCKED
BY FREEZING SCANS
200-340
91.027
± 0.500
100
5:00
200
10:00
300
15:00
400
20:00
500
25:00
600
30:00
700
35:00
SCAN
TIME


MASS CHROMATOGRAMS
12/08/82 16:00:00
SAMPLE: STJACQUES HC-66
CONDS.: 30M SE54,-20 DEG
RANGE: G 1, 750 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
DATA: WH66 #1
CALI: C8121 #1
SCANS 10 TO 750
FIGURE IV
89.4
51
R FREONS & HALON
41728.
51.015
± 0.500
100.0
61
63
VINYL CHLORIDE
DICHLOROETHYLENES
46656.
60.518
62.519
34.0
69
15872.
69.021
± 0.500
38.5
85
17952.
85.025
± 0.500
100
5:00
200
10:00
300
15:00
400
20:00
500
25:00
600
30:00
700
35:00
SCAN
TIME